FINAL BASELINE ECOLOGICAL RISK ASSESSMENT WORK PLAN & SAMPLING AND ANALYSIS PLAN

FOR THE
GULFCO MARINE MAINTENANCE
SUPERFUND SITE
FREEPORT, TEXAS

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AST – above ground storage tank

AVS/SEM – Acid Volatile Sulfide/Simultaneously Extracted Metals

BERA – Baseline Ecological Risk Assessment

CAS – Columbia Analytical Laboratory

COC – chain of custody

COPEC – contaminants of potential ecological concern

CSM – conceptual site model

DDT – dichlorodiphenyltrichloroethane

DQO – Data Quality Objective

EDD – electronic data deliverable

Eh – redox potential

EPA – United States Environmental Protection Agency

FSP – field sampling plan

HPAH – high-molecular weight polynuclear aromatic hydrocarbon

HQ – hazard quotient

LCS – laboratory control sample

LCSD - laboratory control sample duplicate

LPAH – low-molecular weight polynuclear aromatic hydrocarbon

MQL – method quantitation limit

MS – matrix spike

MSD – matrix spike duplicate

NPL – National Priorities List

PAH – polynuclear aromatic hydrocarbon

QAPP – Quality Assurance Project Plan

RI/FS – Remedial Investigation/Feasibility Study

RPD – relative percent difference

SAP – Sampling and Analysis Plan

SLERA – Screening-Level Ecological Risk Assessment

SMDP – Scientific Management Decision Point

SOW – Statement of Work

TCEQ – Texas Commission on Environmental Quality

TOC – total organic carbon

UAO – Unilateral Administrative Order

USFWS – United States Fish and Wildlife Service

1.0 INTRODUCTION

The United States Environmental Protection Agency (EPA) named the former site of Gulfco Marine Maintenance, Inc. in Freeport, Brazoria County, Texas (the Site) to the National Priorities List (NPL) in May 2003. The EPA issued a modified Unilateral Administrative Order (UAO), effective July 29, 2005, which was subsequently amended effective January 31, 2008. The UAO required Respondents to conduct a Remedial Investigation and Feasibility Study (RI/FS) for the Site. Pursuant to Paragraph 37(d)(x) of the Statement of Work (SOW) for the RI/FS, included as an Attachment to the UAO, a May 3, 2010 Final Screening Level Ecological Risk Assessment (SLERA) was prepared for the Site (PBW, 2010a). The Scientific/Management Decision Point (SMDP) provided in the Final SLERA concluded that the information presented therein indicated a potential for adverse ecological effects, and a more thorough assessment was warranted. This Final Baseline Ecological Risk Assessment (BERA) Work Plan & Sampling and Analysis Plan has been prepared, consistent with Paragraphs 37(d)(xi) and (xii) of the UAO as the next step in that assessment. This report was originally prepared by Pastor, Behling & Wheeler, LLC (PBW, 2010b), on behalf of LDL Coastal Limited LP (LDL), Chromalloy American Corporation (Chromalloy) and The Dow Chemical Company (Dow), collectively known as the Gulfco Restoration Group (GRG). This June 22, 2010 revision has been prepared by URS Corporation (URS) based on comments received from the EPA and the Texas Commission on Environmental Quality (TCEQ).

1.1 REPORT PURPOSE

Following completion of the SLERA, the BERA Problem Formulation was conducted to identify the specific ecological issues at the Site and determine the scope and goals of the BERA in accordance with Paragraph 37(d)(xi) (Step 3) of the SOW for the RI/FS. The BERA Problem Formulation further refined or identified contaminants of ecological concern, ecological effects of contaminants, fate and transport, assessment endpoints, and the Conceptual Site Model (CSM). The CSM was used to develop an investigation plan and establish the data requirements and data quality objectives to be achieved through the BERA. This Work Plan has been prepared to describe the CSM and the investigation components necessary to complete the BERA. The Work Plan includes a Sampling and Analysis Plan (SAP) that establishes the specific sampling locations, equipment, and procedures to be used during the BERA.

Per EPA direction, this Final BERA Work Plan and SAP is being submitted concurrent with theJune 22, 2010 Final BERA Problem Formulation Report (URS, 2010). As such, the investigation activities proposed herein may be subject to revision based on review comments and revisions to the Final BERA Problem Formulation Report. Also, a Removal Action Work Plan has been finalized and is ready to be implemented upon execution of the Removal Action Settlement Agreement. This Removal Action is intended to: (1) address the aboveground storage tank farm (AST Tank Farm) in the South Area of the Site; and (2) facilitate repair of the existing cap on the former surface impoundments in the North Area of the Site. If approved, implementation of the removal action in the North Area obviates the need for further consideration of soil exposure pathways through the BERA. Also, as described in the Final BERA Problem Formulation, the South Area does not contain complete exposure pathways relevant to this assessment and is not considered further in the BERA process. The South Area is characterized by the following habitat-related considerations:

1. It is zoned by the City of Freeport as "W-3, Waterfront Heavy", which provides for commercial and industrial land use, primarily port, harbor, or marine-related activities. Since the Site was developed in the early 1960s, it has been used for industrial purposes. It is also bounded by former and/or current industrial properties to the east and west;
2. A restrictive covenant placed on the deed ensures that future land use for this parcel of land is commercial/industrial. The Site will most certainly be used in the future for industrial purposes since the barge slips are valuable to many types of businesses in the area, and it is very unlikely that any portion of the Site will return to "natural" conditions;
3. The South Area does not serve as valuable habitat, foraging area, or refuge for ecological communities, including threatened/endangered or otherwise protected species. The Site has not been used since approximately 1999 and opportunistic grasses and small shrubs have grown on some portions of the Site that do not have concrete, oyster shell, or gravel cover;
4. The South Area does not contain consistent and contiguous habitat but, rather, the area is broken up by the presence of concrete slabs, pads, and driveways;
5. The South Area only exhibits minimal natural functions because of the disturbed nature of the land due to the industrial use of the property and adjacent properties; and
6. There are minimal if any attractive features at the South Area that would support a resident wildlife community.

The objective of this Work Plan and SAP is to document the decisions and evaluations made during the BERA Problem Formulation and to identify the additional investigation activities needed to complete the evaluation of ecological risks. This Work Plan and SAP presents the conclusions of the Final BERA Problem Formulation, and the methods and procedures necessary to complete the BERA based on those conclusions. This Work Plan and SAP includes the general scope of activities to be conducted during the BERA, and a detailed description of the sampling and data-gathering procedures.

1.2 SITE BACKGROUND

The Site is located in Freeport, Texas at 906 Marlin Avenue (also referred to as County Road 756) (Figure 1). The Site consists of approximately 40 acres along the north bank of the Intracoastal Waterway between Oyster Creek (approximately one mile to the east) and the Texas Highway 332 bridge (approximately one mile to the west). The Site includes approximately 1,200 feet (ft.) of shoreline on the Intracoastal Waterway, the third busiest shipping canal in the US (TxDOT, 2001) that, on the Texas Gulf Coast, extends 423 miles from Port Isabel to West Orange.

Marlin Avenue divides the Site into two primary areas (Figure 2). For the purpose of descriptions in this report, Marlin Avenue is approximated to run due west to east. The property to the north of Marlin Avenue (the North Area) consists of undeveloped land and closed surface impoundments, while the property south of Marlin Avenue (the South Area) was developed for industrial uses with multiple structures, a dry dock, sand blasting areas, an aboveground storage tank (AST) tank farm, and two barge slips connected to the Intracoastal Waterway.

Adjacent property to the north, west, and east of the North Area is undeveloped. Adjacent property to the east of the South Area is currently used for industrial purposes while to the west the property is currently vacant and previously served as a commercial marina. The Intracoastal Waterway bounds the Site to the south. Residential areas are located south of Marlin Avenue, approximately 300 feet west of the Site, and 1,000 feet east of the Site.

The South Area includes approximately 20 acres of upland that was created from dredged material from the Intracoastal Waterway. The two most significant surface features within the South Area are a Former Dry Dock and the AST Tank Farm. The remainder of the South Area

surface consists primarily of former concrete laydown areas, concrete slabs from former Site buildings, gravel roadways and sparsely vegetated open areas with some localized areas of denser brush vegetation, particularly near the southeast corner of the South Area.

Some of the North Area is upland created from dredge spoil, but most of this area is considered wetlands, as per the United States Fish and Wildlife Service (USFWS) Wetlands Inventory Map (USFWS, 2008). This wetland area generally extends from East Union Bayou to the southwest, to the Freeport Levee to the north, to Oyster Creek to the east (see Figure 1). The most significant surface features in the North Area are two ponds (the Fresh Water Pond and the Small Pond) and the closed former surface impoundments. The former surface impoundments and the former parking area south of the impoundments and Marlin Avenue comprise the vast majority of the upland area within the North Area.

Field observations during the RI indicate that the North Area wetlands are irregularly flooded with nearly all of the wetland area inundated by surface water that can accumulate to a depth of one foot or more during extreme high tide conditions, storm surge events, and/or in conjunction with surface flooding of Oyster Creek northeast of the Site. Due to a very low topographic slope and low permeability surface sediments, the wetlands are also very poorly draining and can retain surface water for prolonged periods after major rainfall events. Under normal tide conditions and during periods of normal or below normal rainfall, standing water within the wetlands (outside of the two ponds discussed below) is typically limited to a small, irregularly shaped area immediately north of the Fresh Water Pond and a similar area immediately south of the former surface impoundments. Both of these areas can be completely dry, as was observed in June 2008. As such, given the absence of any appreciable areas of perennial standing water, the wetlands are effectively hydrologically isolated from Oyster Creek, except during intermittent, and typically brief, flooding events.

The Fresh Water Pond is approximately 4 to 4.5 feet deep and is relatively brackish (specific conductance of approximately 40,000 umhos/cm and salinity of approximately 25 parts per thousand). This pond appears to be a borrow pit created by the excavation of soil and sediment as suggested by the well-defined pond boundaries and relatively stable water levels. Water levels in the Fresh Water Pond are not influenced by periodic extreme tidal fluctuations as the pond dikes

preclude tidal floodwaters in the wetlands from entering the pond, except for extreme storm surge events, such as observed during Hurricane Ike in September 2008.

The Small Pond is a very shallow depression located in the eastern corner of the North Area. The Small Pond is not influenced by daily tidal fluctuations and behaves in a manner consistent with the surrounding wetland, i.e., becomes dry during dry weather, but retains water in response to and following rainfall and extreme tidal events. Water in the Small Pond is less brackish based on specific conductance (approximately 14,000 umhos/cm) and salinity (approximately eight parts per thousand) measurements.

1.3 REPORT ORGANIZATION

This Work Plan and SAP has been organized in a manner consistent with the recommendation presented in the EPA guidance for conducting ecological risk assessments (EPA, 1997), which is based on the EPA guidance for risk assessments and the EPA guidance for conducting RI/FS studies under CERCLA. A discussion of the Site presented in Section 1. Section 2 presents the Work Plan, including the Conceptual Site Model (CSM), assessment endpoints, risk questions and testable hypotheses, and measurement endpoints. An overview of the ecological investigation design, including the data quality objectives established for the study, are presented in Section 3. The Field Sampling Plan (FSP), which details the sampling types and objectives, sampling location, timing, and frequency, sample designation, sampling equipment and procedures, and sample handling, is presented in Section 4. The Quality Assurance Project Plan (QAPP) is included as Section 5. Health and safety procedures are discussed in Section 6.

2.0 WORK PLAN

2.1 CONCEPTUAL SITE MODEL

Preliminary CSMs for the aquatic and terrestrial ecosystems were described in the SLERA. During problem formulation, these CSMs were updated to consider the results of the contaminants of potential ecological concern (COPEC) refinement, expanded review of potential ecological effects of those COPECs, and the more detailed fate and transport evaluation. Updated CSMs based on these considerations are shown on Figures 3 and 4. These CSMs are discussed below.

The identification of potentially complete exposure pathways is performed to evaluate the exposure potential as well as the risk of effects on ecosystem components. In order for an exposure pathway to be considered complete, it must meet all of the following four criteria (EPA, 1997):

- A source of the contaminant must be present or must have been present in the past.
- A mechanism for transport of the contaminant from the source must be present.
- A potential point of contact between the receptor and the contaminant must be available.
- A route of exposure from the contact point to the receptor must be present.

Exposure pathways can only be considered complete if all of these criteria are met. If one or more of the criteria are not met, there is no mechanism for exposure of the receptor to the contaminant. Potentially complete pathways are shown in the conceptual site models for the terrestrial and estuarine ecosystems (Figures 3 and 4, respectively).

In general, biota can be exposed to chemical stressors through direct exposure to abiotic media or through ingestion of forage or prey that have accumulated contaminants. Exposure routes are the mechanisms by which a chemical may enter a receptor's body. Possible exposure routes include 1) absorption across external body surfaces such as cell membranes, skin, integument, or cuticle from the air, soil, water, or sediment; and 2) ingestion of food and incidental ingestion of soil, sediment, or water along with food. Absorption is especially important for plants and aquatic life.

The terrestrial ecosystem CSM (Figure 3) begins with historical releases of the COPECs from the former surface impoundments and operations areas in the North and South Areas. Soil became contaminated with the COPECs and contaminated soil was transported from its original location to other portions of the Site via the transport mechanisms of surface runoff and airborne suspension/deposition. The significant potential receptors (soil invertebrates) are then exposed to soils in their original location or otherwise via direct contact or ingestion of soil. As previously discussed in Section 1.1, implementation of the removal action in the North Area, as well as the nature of the disturbed habitat in the South Area and past, current, and anticipated future land use (including restrictive covenants for only commercial/industrial land use), obviates the need for further consideration of soil exposure pathways.

The aquatic ecosystem CSM (Figure 4) begins with historical releases of the COPECs from barge cleaning operations that impacted sediment in the barge slips of the Intracoastal Waterway and surface water and sediment in the North Area wetlands. These areas were impacted via the primary release mechanisms of direct discharge from past operations, surface runoff, and particulate dust/volatile emissions. Tidal flooding and rainfall events created secondary release mechanisms of resuspension/deposition, bioirrigation, and bioturbation, such that other areas of surface water and sediment became contaminated. The significant potential receptors (sediment and water-column invertebrates) are then exposed to the contaminated surface water and sediment in their original location or otherwise via direct contact or ingestion of surface water and sediment. The Final SLERA (PBW, 2010a) concluded that there are no unacceptable risks to upper trophic level receptors in any of the aquatic areas.

2.2 ASSESSMENT ENDPOINTS

Assessment endpoints are explicit expressions of the ecological resource to be protected for a given receptor of potential concern (EPA, 1997). Assessment endpoints were identified in the SLERA to focus the screening evaluation on sensitive and susceptible receptors rather than attempting to evaluate risks to all potentially affected ecological receptors. As part of the problem formulation, these assessment endpoints were further refined. The site-specific assessment endpoints are presented in Section 5 of the Problem Formulation and included in Table 1 of this Work Plan.

2.3 RISK QUESTIONS

Ecological risk questions are proposed regarding assessment endpoints and their response to COPECs. These questions are used to guide the study design, evaluate the study results, and perform the risk characterization (EPA, 1997). Risk questions are posed for the assessment endpoints established for the BERA, as presented in the BERA problem formulation and are listed below:

1. Does exposure to COPECs in soil adversely affect the abundance, diversity, productivity and function of the soil invertebrate community? – This risk question is not addressed through this assessment but is mitigated by the proposed remedial action, as previously discussed.
2. Does exposure to COPECs in sediment and surface water adversely affect the abundance, diversity, productivity and function of the benthic invertebrate community?
3. Does exposure to COPECs in sediment and surface water adversely affect the abundance, diversity, productivity and function of the fish community?

2.4 MEASUREMENT ENDPOINTS

The definition of measurement endpoints has evolved over time to include measures of ecosystem characteristics, life-history considerations, exposure, or other measures and is now more accurately termed "measures of effect" (EPA, 1998). The EPA has established three categories of measures:

(1) Measures of effect – Measureable changes in an attribute of an assessment endpoint or its surrogate in response to a stressor to which it is exposed (formerly measurement endpoints);

(2) Measures of exposure – Measures of stressor existence and movement in the environment and their contact or co-occurrence with the assessment endpoint; and

(3) Measures of ecosystem and receptor characteristics – Measures of ecosystem characteristics that influence the behavior and location of entities selected as the assessment endpoint, the distribution of a stressor, and life-history characteristics of the assessment endpoint or its surrogate that may affect exposure or response to the stressor.

Measures of effect and measures of exposure will be used as the measurement endpoints to determine if adverse impacts are potentially occurring to the chosen assessment endpoints. The measure of exposure will be analytical measurements of the COPECs in sediment (bulk and pore water) and surface water samples. The measure of effect will be laboratory toxicity testing of Site samples of bulk sediment and surface water compared to laboratory control samples. Table 1 presents the guilds and their representative receptors, the BERA assessment endpoints, the ecological risk questions and testable hypotheses, the measurement endpoints, and the proposed toxicity tests.

2.5 UNCERTAINTIES AND ASSUMPTIONS

Risk assessments are designed to evaluate uncertainty, which is used to develop an investigation program that will result in the greatest decrease in uncertainty. The principal uncertainties inherent in all risk assessments are identified by the EPA as variability, uncertainty of the true value (i.e., measurement error), and data gaps (EPA, 1998). Throughout the risk assessment process, iterative steps are taken to reduce the uncertainty of the assessment, primarily through the collection of additional data until sufficient evidence has been collected that the inherent uncertainty is reduced to an acceptable level. The approach used in this risk assessment reduces uncertainty by focusing the investigation goals on the specific pathways and receptors identified in the Problem Formulation.

2.5.1 Uncertainties in the Conceptual Site Model

The conceptual model prepared for a site can be the source of significant uncertainty in a risk assessment due to a variety of factors, including lack of knowledge about ecosystem functions, a poor understanding of temporal and spatial parameter interaction, omission of stressors, or neglecting secondary effects (EPA, 1998). The uncertainties in the conceptual model prepared for the BERA have been reduced through the consideration of alternate models that account for a multitude of variables present at the Site.

2.5.2 Uncertainties in the Field Study

Sources of uncertainty in the field study are related to the accuracy of test measurements, the appropriateness of media, sampling, and testing protocols, and the proper selection of sampling

locations. Through strict adherence to the guidelines put forth in the Sampling and Analysis plan, uncertainty associated with the results of the field study will be sufficiently reduced such that the data is legally and scientifically defensible. Measures implemented to ensure this level of data quality include adherence to quality assurance guidelines designed to meet the project DQOs, inclusion of sampling and analysis methods that are well established and accepted in risk assessments, performance of the investigation by appropriately skilled project staff, and multiple checks on data quality prior to use in the risk assessment (i.e., third-party data validation, peer review). The data generated by the field study will represent the Site conditions during a specific time period and does not consider changes in COPEC concentrations, bioavailability, or COPEC sequestration due to temporal effects.

2.5.3 Assumptions

The principal assumption of the field study is that the lines of evidence generated by the field study will be sufficient to satisfy the assessment endpoints and that the data will be an adequate indicator of toxicity associated with COPECs present in the Site sediments. The uncertainty related to these assumptions is based on several factors, including the limitations of the test protocols in identifying effects caused by specific COPECs, toxicity effects due to environmentally modified or biotransformed compounds, and other variables that are not understood using currently available technology.

Other assumptions include:

- The results of the toxicity testing will be indicative of the effects of the COPECs;
- The pore water analytical results are representative of bioavailability;
- Bulk sediment analytical results coupled with TOC and AVS/SEM analyses are representative of bioavailability; and
- Differences in results between reference samples and target samples are a result of differences in chemical concentrations or bioavailability in the media.

3.0 STUDY DESIGN

This section discusses the BERA study design. The study design involves selecting compounds, media, and organisms to be analyzed at the target and reference stations.

3.1 DATA QUALITY OBJECTIVES

Data Quality Objectives (DQOs) were established for the BERA through the Problem Formulation steps, which used the conceptual model to identify the assessment endpoints and risk questions identified in Table 1.

As noted in Section 1.0, the overall objective to be addressed by the BERA is to evaluate the specific contaminants, pathways, and receptors identified in the SLERA as warranting additional investigation. DQOs are based on the proposed end uses of data generated from sampling and analytical activities. DQOs are qualitative and quantitative statements that outline the decision-making process and specify the data required. DQOs are typically developed through a seven-step process (EPA, 2006). However, the DQO development process for ecological risk assessments is constrained by several factors, including the lack of specific criteria for ecological endpoints, the potential for multiple endpoints, and the use of weight-of-evidence evaluations of different measurement types (e.g., contaminant concentrations, bioassay tests). Given these limitations, the steps of the DQO process have been completed in a manner to produce qualitative and quantitative statements to develop an appropriate study design to address the needs of the BERA while still following the 7 steps of the DQO process.

3.2 STATE THE PROBLEM

As noted in Section 1.0, the overall objective to be addressed by the BERA is to evaluate the specific contaminants, pathways, and receptors identified in the SLERA as warranting additional investigation. The objective of this Work Plan and SAP is to document the decisions and evaluations made during the Final BERA Problem Formulation and to identify the additional investigation activities needed to complete the evaluation of ecological risks.

The CSM presented in Section 2.1 of this Work Plan presents the primary release mechanisms, the secondary sources, the secondary release mechanisms, the exposure mediums, the potential

receptors, and the potential exposure pathways to be investigated. The CSM allows for planning to achieve the goals of the study by focusing the investigation.

The planning team members or stakeholders involved in the planning and execution of this SAP include decision makers (e.g., regulating agencies), the responsible parties, as well as those responsible for execution of the project (the contractors). Other people and organizations also may have concerns regarding how the BERA sampling investigation is ultimately executed. In such instances, the decision makers will represent these respective parties and consult with them regarding their concerns and issues.

=Sample collection, toxicity testing, analysis, and data validation following receipt of EPA approval of the Final BERA Work Plan and SAP and is scheduled to be completed in sixty (60) calendar days.

3.3 IDENTIFY THE GOALS OF THE STUDY

These objectives lead to the following three questions or goals of the study.

1. Does exposure to COPECs in soil adversely affect the abundance, diversity, productivity, and function of the soil invertebrate community?
2. Does exposure to COPECs in sediment and surface water adversely affect the abundance, diversity, productivity and function of the benthic invertebrate community?
3. Does exposure to COPECs in sediment and surface water adversely affect the abundance, diversity, productivity and function of the fish community?

3.4 IDENTIFY INFORMATION INPUTS

To address the BERA objectives, an investigation program has been developed to use multiple lines of evidence including sediment toxicity testing, surface water toxicity testing, measures of COPEC bioavailability, and COPEC concentration data.

The investigation program includes bioassays of invertebrates coupled with chemical analyses of soil, sediment, pore water, and surface water. The bioassays, chemical analyses, and

determination of COPEC bioavailability represent three lines of evidence which will be used to support the conclusions of the BERA. The analyses have been selected to incorporate the media, pathways, and COPECs relevant to the assessment endpoints. Sampling, analysis, and data evaluation protocols have been selected to ensure that the data collected is scientifically defensible and applicable to the BERA objectives. Columbia Analytical Services (CAS) has been selected as the analytical laboratory of choice based upon their experience and expertise in analyzing samples in a marine environment, including acid volatile sulfides/simultaneously extracted metals (AVS/SEM). (See Statement of Qualifications presented as Appendix A.)

Samples of bulk sediment and soil for chemical analyses and bioassays, and pore water samples collected for chemical analyses, will be co-located and collected concurrently. Sample station locations have been selected based on COPEC concentrations along a gradient as shown on Table 2. Proposed sampling locations are provided on Figures 5 through 9, and the selection rationale provided in Section 3.5. It should be noted that collection of the amount of pore water required for PAH and pesticide analysis (minimum 2 liters [L] and preferably 4 L) may be difficult. Smaller sample size will result in increased detection limits.

3.4.1 Bioassays

Toxicity analyses will be performed on soils, wetland and estuarine sediments, and estuarine surface water using standard bioassay techniques. The goal of the bioassays will be to quantitatively assess ecological and biological impacts related to the COPECs found in soil, sediment, and surface water at the Site. Sediment bioassay tests will be performed using invertebrates which are intimately associated with soils and sediments due to their burrowing activity or consumption of particulates. Sediment samples collected for bioassay analyses will be co-located and collected concurrently with sediment samples and sediment pore water collected for chemical analyses to ensure correlation among the data. Soil samples will be co-located and collected concurrently with soil collected for chemical analysis. Reference samples will be collected from un-impacted areas to serve as controls for the bioassay analyses. Chronic bioassays utilizing both amphipods and polychaetes have been selected for the sediment and earthworms for the soil. The 28-day chronic bioassay using the amphipod *Leptocheirus plumulosus* and the 28-day chronic bioassay using the polychaete *Neanthes arenaceodentata* have been selected as the most appropriate method for evaluating the sediment toxicity at the Site. The

28-Day chronic bioassay using the earthworm *(Eisenia fetida)* has been selected as the most appropriate method for evaluating soil toxicity at the site.

Leptocheirus plumulosus was selected because this species is representative of the common anthropods found in Texas gulf coast bay systems, and because long-term bioassay information is available. The *Leptocheirus* bioassay tests will use growth, mortality, and reproduction as measurement endpoints. *Neanthes arenaceodentata* were selected because they burrow and ingest sediment which represents significant exposure potential, and they represent one of the most abundant groups of benthic organisms found on the Texas gulf coast. The growth endpoint will be used for this study, with mortality data used only to assist in growth calculations. Both test organisms are sensitive to the Site COPECs, tolerant to a wide range of sediment and salinity conditions, and have been used extensively in bioassay tests.

The sampling depth for sediment will be the top 6 inches. The zone of exposure is relevant to the natural burrowing habits of this type of organism. There are many species within the Genus Neanthes. Burrow depth of the worm can vary by species, location, sediment type, and conditions, but reported depths are generally in the range of 3 to 8 inches (8 to 20 cm). *Neanthes lighti* occupy Y-shaped burrows extending 5-8 inches (12.7-20.3 cm) into sediment, although the worms have been found as deep as 18 inches (45.7 cm) in areas with dropping water levels (Smith, 1953). Hines and Comptois (1985) reported that individuals of Neanthes scuccinea occurred primarily deeper than 2 inches (5 cm), with peak abundance between 3.9-5.9 inches (10-15 cm). According to Sayama and Kurihara (1983), *Neanthes japonica* live in U-shaped burrows having a depth of 3.1 to 3.9 inches (8 to 10 cm).

Surface water toxicity at the Site will be evaluated through the use of a 7-day chronic bioassay analysis that measures survival and growth of *Mysidopsis bahia*. This bioassay was selected based on the appropriateness of the organism for site conditions and the sensitivity of the organism to the COPEC, copper. *Mysidopsis bahia* is more susceptible to exposure to COPECs than fish. Assessing for this receptor is therefore also protective for fish.

Test procedures for the bioassay analyses discussed in this section are provided in Appendix B.

3.4.2 Chemical Analysis

Sediment chemical analysis

Sediments collected as part of the BERA investigation will be analyzed for Site COPECs, grain size, AVS/SEM, and Total Organic Carbon (TOC). According to the EPA guidance document *Contaminated Sediment Remediation Guidance for Hazardous Waste Sites* (EPA, 2005a) concentrations of bulk (total dry weight basis) metals in sediment alone are typically not good measures of metal toxicity. The toxicity of metals can be estimated based on the bioavailable metal fraction, which can be measured in pore water and/or predicted based on the relative sediment concentrations of AVS/SEM and TOC. Both AVS and TOC are capable of sequestering and immobilizing a range of metals in sediment. AVS/SEM analysis will not be performed at Intracoastal Waterway sampling locations since no metal concentrations in Intracoastal Waterway sediments resulted in HQs greater than one. TOC will be measured at all sediment sample locations.

Soil chemical analysis

Soils from the North Area will be analyzed for site COPECs and TOC. Table 2 lists the COPECs and analysis.

Sediment pore water analysis

Sediment pore water will be analyzed for the COPECs indicated on Table 2 and will correspond to the COPECs of interest.

Sediment physical properties analysis

The physical properties of Site sediments were evaluated as part of the RI/FS investigation conducted in 2006. The findings of the RI/FS (report pending) indicate consistent sediment grain size distribution throughout the investigation area. However, grain size will be evaluated at all sediment locations where AVS/SEM analysis is to be conducted.

Surface water analysis

Surface water samples will be analyzed for metals and total acrolein using EPA methods 6010/6020 and 8260, respectively as indicated on Tables 2.

3.4.3 Field Measurements

The following water quality parameters will be measured with a multi-probe sonde at all surface water and sediment sampling locations:

- pH;
- conductivity;
- temperature;
- salinity; and
- dissolved oxygen.

Field measurements of the redox potential (Eh) of sediments will be measured with a portable pH/Eh meter. In addition, field observations of the sediment will be documented, including the sediment texture and consistency; color; presence of biota or debris; and changes in sediment characteristics with depth.

3.5 DEFINE THE BOUNDARIES OF THE STUDY

During the problem formulation step, hazard quotients greater than one for soil invertebrates were calculated for two organic compounds at soil sample location SB-204 in the North Area. The COPECs 4,4'-DDT and Aroclor-1254 had hazard quotients of 9 and 3, respectively, in a sample from this location. This sample location is located south of the former surface impoundments.

Sample locations, rationale, and analytical parameters are presented in Table 2. These locations were selected based upon the results of the Final SLERA (PBW, 2010a) and will serve to address the questions presented in Section 3.3.

Target COPECs selected for the field study were chosen based on the results of the Final BERA Problem Formulation (URS, 2010), which identified the COPECs most likely to cause ecological degradation. Locations represent a cross section of target COPECs and geographic settings across the areas. Sample locations were based ona gradient of COPEC concentrations. Table 2 summarizes the proposed sample locations and analyses. Reference sample locations were selected to be representative of un-impacted Site conditions. Specific sample locations and rationale for selection are presented in Section 4.2 and summarized on Table 2.

3.6 DEVELOP THE ANALYTICAL APPROACH

The chemical concentration data will be evaluated against the toxicity endpoint findings. The bioassay information will be evaluated against relevant ecological endpoints such as mortality and growth. The data will be evaluated to see if there is a correlation between chemical concentration and ecological endpoints. The chemical concentrations and ecological endpoints of the study data will be evaluated against the background/reference locations to determine if there is a difference between those locations and an influence of site related contaminants. If the site-related contaminants show persistent toxicity to the invertebrates indicating a significant risk to the community, then the risk managers would evaluate the practicability of Remedial Actions.

Data generated during the site investigation and analysis phase of the BERA will be used to characterize risk in relationship to the assessment endpoints established in the Problem Formulation. Risks to the assessment endpoints will be determined using a lines-of-evidence approach as described in *Guidelines for Ecological Risk Assessment* (EPA, 1998). During this process, each factor will be carefully examined and evaluated for its importance in characterizing risk assessment endpoints. This approach to risk analysis will rely on quantitative methods of evaluating the measures established for the investigation, including statistical analysis and comparison of data to media toxicity benchmark values.

Bioassay tests will be performed by an experienced and accredited laboratory with appropriate replicates and quality control measures to ensure strong statistical reliability and accuracy of test results. Quality control measures will be documented and later included as an appendix to the BERA. Bioassay test results will be compared to the results obtained from reference samples collected from the same media near the Site. Bioassay results will also be compared to laboratory control samples. The performance of the reference sample bioassays will be used as a control measure to distinguish between toxicological effects likely caused by Site COPECs or toxicological effects resulting from environmental factors (naturally occurring site conditions or laboratory environment). Following validation of the bioassay results and incorporation of reference sample impacts, bioassay data will be evaluated against other applicable lines of evidence, such as bioavailability and concurrently measured COPEC concentrations, to derive statements that are appropriate to address the assessment endpoints.

Chemical analysis of interstitial water and bulk sediment, as well as TOC and AVS/SEM, will be evaluated using established techniques (e.g., equilibrium partitioning) to determine the site-specific bioavailability of Site COPECs. The bioavailability characteristics of the COPECs will be further refined through the use of a literature search to ensure they are applied appropriately. COPEC bioavailability will be incorporated into the overall assessment of the investigation results and conclusions of risk characterization later in the BERA.

COPEC concentrations in environmental media (i.e., surface water, sediment) will be used to correlate bioassay and bioavailability results to toxicological effects, or lack thereof, of specific COPECs. Concentration data will be used to establish hazard quotient values necessary to evaluate ecological risk at the Site.

3.7 SPECIFY ACCEPTANCE CRITERIA AND TOLERABLE LIMITS ON DECISION ERROR

The objective of this step is to specify the quantitative limits that will be used with the decision rule discussed in Section 6.0. These criteria will identify potential error in the decision-making process and the means by which error will be minimized to acceptable levels. The three steps of the process are as follows:

1. Identify types of decision errors and associated impacts;
2. Identify ways to minimize error; and
3. Identify how error will be quantified and assessed.

3.7.1 Types of Decision Errors

There are two types of decision errors: Type I (false rejection of the null hypothesis) and Type II (false acceptance of the null hypothesis).

- *Type I, False Rejection* - This error is the belief that for the P^{th} percentile there is no increase in adverse effect between the concentration and the control when in reality the P^{th} percentile does have an adverse effect. The consequence of this type of error is that adverse effects are present at the site. In other words, a site is concluded to be clean when in reality it remains contaminated.
- *Type II, False Acceptance* - This error is the belief for the P^{th} percentile there is increase in an adverse effect between the concentration and the control, when in reality the P^{th} percentile is less than or equal to the control. The consequence of this type of error is

that resources may be spent unnecessarily to further remediate a site. In other words, a site is concluded to be contaminated when in reality it is clean.

The consequences of such errors depend upon the null hypothesis used when assessing the sites in question. The primary purpose for sampling (i.e., the working hypothesis) is to determine if there is an adverse effect between the concentration and the control. Table 3 shows how these errors relate to statistical level of confidence and power.

A No-Observed-Adverse Effect Level (NOAEL) is the highest exposure level at which no statistically or biologically significant increases are seen in the frequency or severity of adverse effect between the exposed population and its appropriate control population. In an experiment with several NOAELs, the NOAEL is the highest experimentally determined concentration without a statistically or biologically significant adverse effect. In cases in which a NOAEL has not been demonstrated experimentally, the term Lowest-observed-adverse-effect level (LOAEL) is used. The LOAEL is the lowest concentration tested. For this project, the Type 1 error of this hypothesis, α less than 0.05. Therefore, the null hypotheses, there is no increase in the adverse effect between the concentration and control would be rejected if the *P* value is less than 0.05.

Table 3. Summary of Potential Decision Errors

Reality	Conclusion	
	Site Is Contaminated	**Site Is Not Contaminated**
Site Is Contaminated	Correct Probability $\geq 1 - \alpha =$ Level of Confidence	Type I Error False Rejection Probability $< \alpha$
Site Is Not Contaminated	Type II Error False Acceptance Probability $< \beta$	Correct Probability $\geq 1 - \beta =$ Power

3.7.1.1 Minimization of Error

Error is minimized through sample size calculations and the development and implementation of a comprehensive SAP and QAPP. The inputs to the sampling design are set so as to minimize the Type I and Type II decision errors. The SAP and QAPP will be used to provide the foundation for generating quality data with which sound decisions can be made.

In addition to the SAP and QAPP, all analytical data generated will undergo a rigorous review. The review will include, but not be limited to, a validation program.

3.7.2 Non-Random Sampling

Samples will be selected on the basis of knowledge of the site or non-random sampling. For this program, sample locations will be selected on a gradient. The location of the highest concentration of a contaminant will be selected, sampled and analyzed for all COPECs. Then a mid-concentration location will be selected, sampled and analyzed for all COPECs. Finally, a low-concentration location will be selected, sampled and analyzed for all COPECs. In this manner, a gradient will be developed for all COPECs.

Statistical methods may be used to calculate the minimum number of samples needed to estimate the UTL based on predefined values for the Type I and Type II decision errors and the desired percentile. The working hypothesis is set up so that the consequences of a Type I error are more serious than a Type II error because the consequences of the Type I error are that action is not taken when it should be. Therefore, more stringent limits are placed on the Type I error rate (α), while less stringent limits are placed on the Type II error rate (β). Although β is not directly used in the sample size equation (see Section 7.3), it can be minimized by increasing the percentile, *P*. For these sample size calculations, a 95% level of confidence (α=5%) and 90^{th} percentile are used typically used to minimize each type of error. These parameter values are reasonable based on the EPA Soil Screening Guidance User's Guide.

3.7.3 Sampling and Analysis Plan

The objective of this sampling is to develop a gradient of each COPEC with full coverage of the site and to develop toxicity impacts for each COPEC across the site.

3.7.4 Data Validation

All analytical data will be validated. The validation will be conducted in accordance with the SAP.

3.8 DEVELOP THE PLAN FOR OBTAINING DATA

This BERA Work Plan and SAP present the plan for obtaining data.

4.0 FIELD SAMPLING PLAN

4.1 SAMPLING TYPES AND OBJECTIVES

4.1.1 Soil Sampling

Soil sample stations were selected based on investigation requirements and the rationale presented in Section 3. A sample station map will be developed and the sample station coordinates will be determined before sampling is initiated. Soil samples collected from each location for chemical analysis and toxicity testing will be collected at the same time (concurrent and co-located) and at the same depth interval.

Samples will be collected no deeper than two feet. The sample will be collected using a hand-auger and will be placed in a stainless steel bowl for homogenization. Aliquots of the sample will be removed from the bowl and placed in pre-cleaned labeled sample jars. Equipment used for sample collection, sub-sampling, and sample mixing (i.e., spoons, knives, scoops) will be stainless steel or Teflon®.

4.1.2 Sediment Sampling

Sediment sample stations were selected based on investigation requirements and the rationale presented in Section 3.4. A sample station map will be developed and the sample station coordinates will be determined before sampling is initiated. Sediment samples collected from each location for chemical analysis, pore water extraction, and toxicity testing will be collected at the same time (concurrent and co-located) and at the same depth interval.

Sampling will be conducted from a boat, skiff, on foot, or other appropriate sampling platform as conditions indicate. Sampling in areas inaccessible by watercraft will be conducted by wading to the sample stations. A differential GPS receiver with sub-meter accuracy will be used to locate the stations and record actual coordinates, as detailed in Section 4.2. Sample station information, sample depth, and all other pertinent observations made during the study will be recorded on field data sheets. The following sections describe the basic sediment sampling procedures for the various techniques to be employed during the investigation.

Marsh and Wetland Sediment

Sediment will be collected from the intertidal marsh by approaching the sample site on foot, being careful not to impact the area to be sampled. The sample will be collected using a stainless steel scoop or spoon, and will be placed in a stainless steel bowl for homogenization. Aliquots of the sample will be removed from the bowl and placed in pre-cleaned labeled sample jars. Equipment used for sample collection, sub-sampling, and sample mixing (i.e., spoons, knives, scoops) will be stainless steel or Teflon®. Sediment samples collected for AVS/SEM analysis will be collected separately from the other samples (but at the same depth) and transported in a manner specified by the laboratory to reduce the likelihood of exposure to atmospheric conditions.

Intracoastal Waterway Sediment

Soft surficial sediment samples will be collected using an Ekman grab (or equivalent). The jaws of the sampler will be locked open and the sampler will be lowered to the bottom on a cable or attached to a stainless steel pole. To prevent forward wake, the sampler will not be lowered faster than 0.3 m/sec as it nears the bottom. The sampler will be retrieved slowly to ensure proper jaw closure. The retrieved sampler will be lowered into a clean tub or tray, and secured in an upright position to prevent sediment movement. Collection of sediments using an Ekman or Ponar Grab device is also described in SOP-BESI-101 previously provided in the RI/FS Field Sampling Plan (PBW, 2006b).

A sediment sample will be acceptable if its depth is greater than 6 inches and the surface is relatively flat and undisturbed. If a sample is not acceptable it will be set aside (do not dump overboard), and a second sample will be collected. Unacceptable samples will be discharged overboard after an acceptable sample is collected.

Prior to removing sediments from the sampler, overlying water will be drained by gently tilting it. Care will be taken so that fine sediments are not decanted. A 0 to 6-inch sub-sample will be collected from the top of the closed sampler using a pre-cleaned spoon, scoop, or core tube. Sediment will be removed using pre-cleaned spoons and composited in pre-cleaned stainless steel bowls. Only the sediment from the center of the grab sampler (i.e., no sediment touching the walls of the sampler) will be used. Equipment used for sample collection, sub-sampling, and sample mixing (i.e., spoons, knives, scoops) will be stainless steel or Teflon®. Sediment samples

collected for AVS/SEM analysis will be collected and transported in a manner specified by the laboratory to reduce the likelihood of exposure to atmospheric conditions.

Core Sampler

Samples of stiff sediment samples from the Intracoastal Waterway, Fresh Water Pond, and/or Small Pond may be collected using a piston-coring device if the grab sampler is not effective at collecting a representative sample. The coring device consists of a 3-inch diameter polycarbonate core tube attached to the end of an aluminum pole. The coring device will be manually driven into the sediment until firm resistance is detected. In the event that a single core does not provide the volume of material required by the analytical laboratory (approximately 1 liter), additional cores will be collected at that station to provide the required sediment. All cores samples from the same station will be combined and homogenized before aliquots are removed.

Sediment from 0-6 inches will be extruded into a stainless steel bowl and will be homogenized and placed in containers for other analyses.

The empty sampler (Ekman or core) will be rinsed and decontaminated following the procedures presented in Section 5.11. The sampler and associated equipment will be decontaminated before use, and between sample sites. In addition, the sampler will be rinsed with Site water before samples are collected.

4.1.3 Pore Water Sampling

Sediment pore water samples will be co-located with bulk sediment sample stations and will be collected concurrently with bulk sediment samples. Sediment samples collected for pore water analyses will be collected using a piston corer (SOP-BESI-102, RI/FS Field Sampling Plan, PBW, 2006b). Several 2 to 3 ft long core tubes will be collected at each station and the top six inches of sediment will be used for processing. Sediment samples will be kept in the core tube after sampling, capped, and transported to the processing area without disturbing the sediment. Processing will consist of centrifuging aliquots of the sediment samples until the pore water is separated from the sediment. The pore water is removed using a syringe and then filtered into a standard sample container. Due to the difficulty associated with pore water extraction and the

limited volume of pore water generated, some detection limits may be elevated due to limited sample volumes.

4.1.4 Surface Water Sampling

Surface water samples will be collected from one location north of the wetlands north of Marlin Avenue. The surface water sample will be collected from the water surface using a bailer, dip sampler or other discrete depth sampling equipment. Surface water sampling will be conducted in accordance with the SOP provided in the RI/FS Field Sampling Plan (SOP 10, Water Quality Sampling, PBW, 2006b).

4.2 SAMPLING LOCATIONS, TIMING, AND FREQUENCY

Proposed sampling locations are presented on Figures 5 through 9, and summarized on Table 2. The sample locations and rationales for selection are also presented on Table 2.

Locating Proposed Sampling Stations

Sample stations will be located in the field using the coordinates extrapolated from proposed sample locations on the Site maps. A GPS receiver will be used to locate the proposed sampling sites in the field. The GPS unit will utilize real-time corrections to achieve the horizontal coordinates with sub-meter accuracy. Accuracy of the sample locations is important to mapping analytical results, so a relatively high degree of confidence is needed as to where each sample is collected, and if needed, the sample location can be reacquired for future efforts. The desired coordinates will be programmed into the GPS and the receiver can then guide the user to the desired coordinates. However, the proposed sampling locations may be modified in the field based on field conditions and professional judgment. If samples are collected from a sampling vessel, the sampling vessel will be secured at the station using a minimum of two anchors (one placed off the bow and one placed off the stern) to ensure the effects of crosswinds and/or tides are minimized.

Sampling Frequency and Timing

The investigation is planned as a one-time sampling event that will not require additional routine sampling events. The sampling event will be conducted within a reasonable timeframe following approval of the applicable project documents. Depending on the specific analytical methods chosen for the investigation, seasonal influences on bioavailability may be factored into the timing of the sampling event.

There is a sixty (60) calendar day schedule for sample collection, toxicity testing, analysis, and data validation.

4.3 SAMPLE DESIGNATION

The station and sample numbering system for the project has been designed to uniquely identify each sampling station and sample. This numbering system consists of the sample location identifier, depth (if applicable), and QA/QC identifier (if applicable). Sample locations will typically correspond to previous sampling locations that indicated an exceedance during the SLERA.

Sample locations will be designated by the investigation identifier "E" for "ecological risk assessment", followed by a Site location identifier i.e., "W" for wetland, followed by the sample type, i.e., SED, followed by the locations number (1, 2, 3…). Depth intervals in feet below grade will be assigned to sediment samples to designate the vertical sample location. Pore water samples will have the identifier "PW" appended to the sample ID. As an example, a sediment sample collected from 0 to 6 inches deep in the Intracoastal Waterway at sample station No. 1 will be designated as follows:

Sample ID: EIWSED01 (0-6) (sample IDs listed on Table 2)

A sample of pore water collected at this location would be assigned a sample ID of "EIWSED01PW".

Field quality control samples such as matrix spikes and matrix spike duplicates and field duplicates, which are detailed in the QAPP, will be designated with the primary sample identification and a quality control suffix as noted below.

Quality Control	Suffix Description	Sample Frequency
MS/MSD	Matrix spike/duplicate	1 per 20 samples per media
FD	Field duplicate	1 per 20 samples per media
EB	Equipment rinsate blank	1 per day/team
FB	Field blank	1 per day/team

To prevent misidentification of samples, labels will be affixed to each sample container. Information will be written on the label with a permanent marker. The labels will be sufficiently durable to remain legible even when wet and will contain the following information:

- Project identification number;
- Sampling station identification name;
- Name or initials of collector;
- Date and time of collection;
- Analysis required (if space on label allows); and
- Preservative inside bottle, if applicable.

4.4 SAMPLING EQUIPMENT AND PROCEDURES

4.4.1 Field Data, Equipment, and Instrument Calibration

Field data will primarily be direct observations, hand measurements, and direct-readings from field meters. These data will be tabulated and included in project reports or submittals, as appropriate. Appropriate field forms will be used to record field data collection activities.

Samples will be collected following the sampling procedures documented in this FSP. The equipment used to collect samples, time of sample collection, sample description, volume and number of containers, and preservatives added (if applicable) will be recorded on the appropriate field forms.

All field monitoring equipment will be calibrated at the beginning of each day before sample collection and when in use, if necessary. For each meter, recalibration requirements will be based on the manufacturer's guidelines and appropriate SOPs.

A Chain-of-Custody document will be initiated for the samples, and the appropriate information will be recorded on both the field-log sheet and chain document, as detailed in Section 5.4.

4.5 SAMPLE HANDLING

Samples will be preserved as indicated in Section 5 (QAPP), and stored, as necessary, on ice until shipped to the laboratory for analysis. To meet sample holding times, the samples will be packed

in coolers and shipped as soon after collection as practical. Sample volumes, preservative, and holding time requirements are summarized on Table 3.

Samples will be placed in shipping coolers containing bagged, cubed ice immediately following collection. The samples will be grouped in the shipping cooler by the order in which the samples are collected. Samples to CAS will be shipped to the laboratory via an overnight courier service, generally on the day they are collected. The only exceptions to this procedure will be for samples collected after the courier service has picked up the shipment for the day and samples collected on a Sunday or holiday. In these instances, the samples will be shipped on the next business day. Specific protocols are included in PBW SOP-6: *Sample Custody, Packaging and Shipment* provided in the RI/FS Field Sampling Plan (PBW, 2006b). Samples to PBS&J may be transported directly to the lab or shipped via an overnight courier service, as described above.

Evidence of collection, shipment, and laboratory receipt must be documented on a Chain-of-Custody record by the signature of the individuals collecting, shipping and receiving each sample. A sample is considered in custody if it is:

- In a person's actual possession;
- In view, after being in physical possession;
- Sealed so that no one can tamper with it, after having been in physical custody; and/or
- In a secured area restricted to authorized personnel.

Chain-of-Custody Records will be used, by all personnel, to record the collection and shipment of all samples. The Chain-of-Custody Record may specify the analyses to be performed and should contain at least the following information:

- Name and address of originating location of samples;
- Name of laboratory where samples are sent;
- Any pertinent directions/instructions to laboratory;
- Sample type (e.g., aqueous);
- Listing of all sample bottles, size, identification, collection date and time, and preservative, if any, and type of analysis to be performed by the laboratory;
- Sample ID;

- Date and time of sample collection; and
- Signature of collector as relinquishing, with date/time.

The Chain-of-Custody procedure will be as follows:

The field technician collecting the sample shall be responsible for initiating the Chain-of-Custody Record. The names of all members of the sampling team will be listed on the Chain-of-Custody Record. Samples can be grouped for shipment on a common form.

Each time responsibility for custody of the samples changes, the receiving and relinquishing custodians will sign the record and note the date and time.

1) The Chain-of-Custody Record shall be sealed in a watertight container, placed in the shipping container, and the shipping container sealed prior to giving it to the carrier. The carrier waybill shall serve as an extension of the Chain-of-Custody Record between the final field custodian and receipt in the laboratory. The commercial carrier is not considered part of the COC chain and is not required to sign the COC.

2) Upon receipt in the laboratory, a designated individual shall open the shipping containers, measure and record cooler temperature, compare the contents with the Chain-of-Custody Record, and sign and date the record. Any discrepancies shall be noted on the Chain-of-Custody Record.

5) If discrepancies occur, the samples in question shall be segregated from normal sample storage and the project manager will be notified for clarification.

6) Chain-of-Custody Records, including waybills, if any, shall be maintained as part of the project records.

4.6 SAMPLE ANALYSIS

4.6.1 Proposed Laboratories

Bioassay

PBS&J
888 West Sam Houston Parkway South
Suite 110
Houston, TX 77042-1917
713-977-1500

Chemical Analysis

Columbia Analytical Services
1317 South 13th Avenue
Kelso, Washington 98626
360-577-7222

The laboratories chosen to provide analytical services for the BERA were selected based on historical performance and areas of technical expertise related to ecological risk assessments. SOPs for test methods provided by the bioassay laboratory are provided in Appendix B. A Statement of Qualifications and Quality Assurance/Quality Control Manual for PBS&J and CAS are provided in Appendix C.

4.6.2 Chemistry Analysis Methods

Chemistry analyses will be conducted according to established EPA or ASTM methods. The analytical methods selected for use during this investigation are presented in Table 4 and listed below:

- Metals – EPA Method 6010/6020
- PAHs and hexachlorobenzene – EPA Method 8270C
- Organochlorine Pesticides – EPA Method 8081
- PCBs – EPA Method 8082
- TOC – SW846 Method 9060
- AVS/SEM – EPA Draft Analytical Method EPA/821/R-91/100
- Grain Size - ASTM D422

4.6.3 Toxicity Testing Methods

Bioassay tests were selected based on the appropriateness of the test organism relative to the physical characteristics of the Site (salinity, sediment grain size, etc.) and sensitivity to the Site COPECs. The specific species were selected because of their interaction with sediment (burrowing and ingestion), they are representative of one of the most abundant groups of benthic organisms found in Texas bays (polychaetes), they represent one of the most abundant groups of crustaceans found in Texas bays (amphipods), and they have been used extensively in similar ecological assessments. Toxicity tests selected for use in the ecological risk assessment are provided on Table 4 and listed below. The test procedures for bioassay tests are provided in the SOPS included in Appendix B.

Sediment

- 28d chronic (growth, survival, reproduction) bioassay using *Leptocheirus plumulosus; and*
- 28d chronic (growth and survival) bioassay using *Neanthes arenaceodentata.*

Surface water

- 7d chronic (growth and survival) bioassay using *Mysidopsis bahia.*

Soil

- 28 day chronic (growth and survival) bioassay using *Eisenia fetida.*

4.7 CONTINGENCIES

This section describes contingency procedures to be used if a portion (or portions) of the steps described in this Work Plan cannot be performed. Contingency planning includes informing the EPA of problems encountered and alternate actions being considered. The EPA will also be notified of other problems that may be encountered during sample collection and transport, such as sample loss or container breakage.

The type of contingency procedures required (e.g., departures or deviations) will be recorded on field sheets. EPA will be informed of all deviations, considered one-time occurrences, as soon as is practical.

5.0 QUALITY ASSURANCE PROJECT PLAN

5.1 PROJECT DESCRIPTION

This QAPP has been prepared for the BERA at the Gulfco Marine Maintenance Site. The BERA Work Plan that includes this QAPP describes the project background and investigation objectives, including the site description and history, the project objectives, and the sample network design and rationale. The FSP describes procedures to be implemented in the field. Investigation specific procedures and protocols for sample collection, chain-of-custody, sample handling, sample analysis, and report preparation are included in this QAPP or by reference to the previously submitted Sampling and Analysis Plan (SAP) included in the RI/FS Work Plan prepared for the Site (PBW, 2006c). The QAPP is organized in accordance with basic EPA guidelines for the preparation of QAPPs. Laboratory Quality Manuals are presented in Appendix C.

The goal of the QAPP is to assure that the data collected meet the project objectives established in Section 3.1. All QA/QC procedures will be in accordance with applicable professional standards, government regulations and guidelines, and specific project goals and requirements.

5.2 QA/QC ORGANIZATION AND RESPONSIBILITIES

Respondent's Project Coordinator

The Respondent's Project Coordinator will direct and supervise all BERA work. The Project Manager's responsibilities will be to review all BERA project work to ensure that it meets the specific project goals, meets technical standards, and is in accordance with the objectives and procedures discussed herein.

BERA Investigation Manager

The BERA Investigation Manager will direct and supervise all BERA work. The BERA Investigation Manager's responsibilities will be to review all BERA project work to ensure that it meets the specific project goals, meets technical standards, and is in accordance with the objectives and procedures discussed herein.

QA Manager

The QA Manager will remain independent of direct involvement in day-to-day operations, but will have direct access to staff, as necessary, to resolve any QA issues. The QA Manager has sufficient authority to stop work on the investigation as deemed necessary in the event of serious QA/QC issues. Specific functions and duties include:

- Performing QA audits on various phases of the project's operations, as necessary;
- Reviewing and approving this QAPP and other QA plans and procedures;
- Performing validation of data collected relative to risk assessment activities and this QAPP; and
- Providing QA technical assistance to project staff.

The QA Manager will notify the Project Coordinator of particular circumstances that may adversely affect the quality of data and ensure implementation of corrective actions needed to resolve nonconformances noted during assessments.

Field Supervisor

The Field Supervisor will be responsible for all aspects of field work performed as part of a specific risk assessment activity. Different project subtasks or activities may have different Field Supervisors. Duties of the Field Supervisor will include:

- Maintaining field records;
- Continually surveying the Site for potential work hazards and relate any new information to site personnel at the Tailgate Safety Meeting held each day prior to beginning field activities;
- Ensuring that field personnel are properly trained, equipped, and familiar with Standard Operating Procedures and the Health and Safety Plan;
- Overseeing sample collection, handling and shipping; ensuring proper functioning of field equipment; and
- Informing the laboratory when samples are shipped to the lab and verifying samples arrived at the lab.

The primary duty of the Field Supervisor is to ensure that the field sampling is performed in accordance with the project sampling plans and this QAPP. The Field Supervisor will also require that appropriate personal protective equipment will be worn and disposed of according to the Health and Safety Plan provided in the RI/FS SAP prepared for the Site (PBW, 2006b). In addition, the Field Supervisor may be responsible for preparing monitoring reports for review by the Project Manager.

Laboratory QA Manager

The laboratory QA Manager will have overall responsibility for data generated in the laboratory. The laboratory QA Manager will be independent of the laboratory production responsibilities, but will communicate data issues through the Project Manager. In addition, the laboratory QA Manager will

- Monitor the day-to-day quality of the laboratory data;
- Maintain and review all quality control data;
- Conduct internal performance and system audits to ensure compliance with laboratory protocols.;
- Review and maintain updated Standard Operating Procedures (SOPs); and
- Prepare Performance Evaluation reports and corrective action reports.

5.3 PRECISION, ACCURACY, COMPLETENESS, REPRESENTATIVENESS, COMPARABILITY AND SENSITIVITY

Performance objectives have been established for each of the Data Quality Indicators (Precision, Accuracy, Completeness, Representativeness, and Comparability) as defined below.

5.3.1 Precision

Precision is a measure of the reproducibility between two or more measurements of the same characteristic (i.e., analyte, parameter) under the same or similar conditions. Determining the agreement among replicate measurements of the same sample assesses the precision of the analytical procedure; combined precision of sampling and analysis procedures is assessed from the agreement between measurements of field duplicate samples. The relative percent difference

(RPD) in the results will be computed for each duplicate pair. The RPD is defined as 100 times the absolute value of the difference (range) of each duplicate set, divided by the average value (mean) of the set:

$$\text{RPD} = \frac{\text{ABS}(\text{primary sample result} - \text{duplicate sample result})}{\text{average of primary and duplicate sample result}} \times 100$$

Field Precision Objectives

Precision of sampling and analysis procedures will be assessed through the collection of field duplicate samples. Data for duplicate analyses will be evaluated only if both of the samples in the duplicate pair have a concentration greater than the method quantitation limit (MQL). It is noted here that natural variation in some of the matrices will affect how closely these goals are met; that is, if variation is high, then these goals are unrealistic. Consequently, RPD results from field duplicates will not be used as a basis for invalidating any analytical data. The RPD goals for water field duplicates are RPD $\leq$30% and for sediment are RPD $\leq$50%.

Laboratory Precision Objectives

Precision of the analytical procedure will be assessed through duplicate analyses of laboratory QC and field samples. Data for duplicate analyses will be evaluated only if both of the samples in the duplicate pair have a concentration greater than the method quantitation limit (MQL). Precision goals are presented in Table 5.

5.3.2 Accuracy

Accuracy is a measure of the bias in terms of the degree of agreement between an observed value (i.e., sample result) and the accepted reference or true value. Accuracy is expressed as the percent recovery of spiked analytes. The equations used to calculate percent recovery is:

$$\%\ \text{Recovery} = \frac{\text{measured amount}}{\text{known amount}} \times 100$$

Laboratory blank samples and field blanks will also be used to quantify the effect of sample contamination on overall data accuracy.

Field Accuracy Objectives

The potential for field contamination will be assessed through collection of equipment blanks (when non-dedicated sampling equipment is used) and trip blanks (as needed) and adherence to all sample handling, preservation and holding time requirements.

Laboratory Accuracy Objectives

Laboratory accuracy will be evaluated by the analysis of laboratory control samples (LCS), matrix spike (MS) samples and surrogate spikes, with results expressed as a percentage recovery measured relative to the true (known) concentration. In addition, laboratory preparation blank results will be used to measure any contamination introduced during the analytical process. The objectives for minimizing the effect of laboratory contamination on sample accuracy are concentrations less than the MQL in all blank samples. LCS and MS acceptance criteria are presented in Table 5. Data will not be rejected based upon MS recoveries.

5.3.3 Completeness

Completeness is the percentage of valid measurements or data points obtained, as a proportion of the number of measurements or data points planned for the project. Completeness is affected by such factors as sample bottle breakage and acceptance/rejection of analytical results. Completeness will be re-calculated and presented in each validation checklist. If completeness approaches the established goal (within 2-3%), corrective action will be instituted as described in Section 5.9. The completeness goal for soil and sediment samples is sample level is 90% and for water samples is 100%.

5.3.4 Representativeness

Representativeness is a qualitative objective, defined as the degree to which data accurately and precisely represents the characteristic of a population, the parameter variations at a sampling point, the process condition, or an environmental condition within a defined spatial and/or temporal boundary.

Field Representativeness Objectives

Field representativeness is achieved by collecting a sufficient number of unbiased (representative) samples and implementing a QC program for sample collection and handling prior to analyses. The sampling approaches developed for this project will provide for samples that are representative of site conditions. Any equipment blank and field blank results will also be evaluated to ensure that analytical results are representative of sample concentrations.

Laboratory Representativeness Objectives

Representativeness in the laboratory is ensured by using the proper analytical procedures, appropriate sample handling and preparation methods, meeting sample holding times and analyzing and assessing duplicate samples.

5.3.5 Comparability

Comparability is the confidence with which one data set can be compared to another.

Measures to Ensure Comparability of Field Data

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the standard field protocols in the FSP are consistently followed and that the sampling techniques specified in the sampling plan are consistently used.

Measures to Ensure Comparability of Laboratory Data

Planned analytical data will be comparable when the sampling and analytical methods described in the FSP and in this QAPP are used for sample collection and laboratory analysis. This goal is achieved through the consistent use of standard techniques to collect and analyze representative samples. Results of sample analyses will be consistently reported in appropriate units. Comparability is also dependent upon the laboratory obtaining the QA objectives for accuracy and precision. All data that meet the QA objectives described in this document and are considered usable will be considered comparable data.

5.3.6 Sensitivity

Analytical methods have been selected based upon the sensitivity of the method detection limits. To ensure that the data are usable, the method must be able to meet the ecological endpoints. A comparison of laboratory method detection limits and ecological endpoints is presented in Table 6.

5.4 SAMPLING PROCEDURES

Project sampling processes were designed to obtain information necessary to address those data needs described in the CSM, and identified during the BERA Problem Formulation step. Field sampling procedures employed during the ecological risk assessment will be consistent throughout the project, thus providing data representative of site conditions, comparability with analytical considerations, practicality, and simplicity. Procedures for all aspects of collection, preservation, and transport of samples are provided in the FSP.

5.4.1 Sampling Methods

Sampling methods are described in Section 4.0 of this Work Plan. SOPs for these methods are provided in Appendix A of the RI/FS FSP (PBW, 2006b).

Sample Volume, Containers, and Preservation

The sample volume, container and preservation requirements will be in accordance with requirements for the specific analytical methods. This information is provided in Table 3.

5.4.2 Sampling Quality Control Requirements and Acceptability Criteria

Field Duplicate

Field duplicates will be collected for chemical analyses at the frequency of one per 20 field samples collected or at least one per sampling day (excludes bioassay samples). A field duplicate is defined as a second sample (or measurement) from the same location, collected in immediate succession, using identical techniques. The duplicate sample will be collected from the same homogenized composite material as the sample it is duplicating. Duplicate samples are sealed, handled, stored, shipped, and analyzed in the same manner as the primary sample. Precision of

duplicate results is expressed by the RPD between the results of the two samples. Precision goals for sediment samples are RPD ≤50% and for aqueous samples the goal is an RPD ≤30%.

Field Splits

Field splits are not required for any of the activities, but may be requested by the EPA. A field split is collected in the same manner as a field duplicate. Precision goals for sediment samples are RPD ≤50% and for aqueous samples the goal is an RPD ≤30%.

Equipment Blanks

Equipment blanks (rinsate) blanks may be collected when sampling requires the re-use of non-dedicated equipment. If required, equipment blanks will be collected once per day, from decontaminated sampling equipment and analyzed for the COPECs of interest. When possible, rinsate blanks will be collected from the final rinse water of non-dedicated decontaminated equipment to assess the effectiveness of the cleaning and decontamination procedure. Rinsate blanks will be used to qualify the data and may be used to invalidate the sample results.

Trip Blanks

Trip blanks are typically included in sample shipping containers to evaluate the potential for contamination from VOCs during sample transport. Since trip blanks are used only when samples are collected for volatile organic compounds analyses, not all activities will require trip blanks. Trip blanks will be used to qualify the data and may be used to invalidate the sample results.

5.4.3 Field Sample Handling and Custody

Chain-of-Custody (COC)

Proper sample handling and custody procedures ensure the custody and integrity of samples beginning at the time of sampling and continuing through transport, sample receipt, preparation, analysis, and disposal.

A sample is in custody if it is in actual physical possession or in a secured area that is restricted to authorized personnel. The COC form is used to document sample handling during transfer from

the field to the laboratory and among contractors. The list of items below should be included on the COC form.

- Site identification
- Sample identification
- Date and time of collection
- Sample matrix
- Container type
- Number of containers
- Preservative used
- Notation if the sample was filtered
- Analyses required
- Name and signature of collector(s)
- Custody transfer signatures and dates and time of transfer
- Name of laboratory admitting the samples
- Bill of lading (if applicable)

Sample Labeling

Sample labels are completed with an indelible, waterproof marker. Label information includes the sample identification number, the date and time of sampling and sample type. The sample identification numbering system for the project has been designed to uniquely identify each sampling station and sample. This numbering system consists of a sequential sample location identifier, depth (if applicable), and QA/QC identifier (if applicable), as detailed in the FSP.

Sample Handling

Sample handling procedures for each activity and type of sample are described in the FSP.

Failures in Chain of Custody and Corrective Action

All failures associated with COC procedures are immediately reported to the person who originally signed the COC, typically the Field Supervisor. These include such items as delays in transfer, resulting in holding time violations; violations of sample preservation requirements; incomplete documentation, including signatures; possible tampering of samples; broken or spilled

samples, etc. The Project Manager or Field Supervisor, in consultation with the QA Manager, will determine if the procedural violation may have compromised the validity of the resulting data. Any failures that have reasonable potential to compromise data quality will invalidate data, and the sampling event should be repeated. The resolution of the situation will be reported to the Project Coordinator. Corrective action reports will be maintained by the QA Manager.

5.4.4 Laboratory Sample Handling and Custody

Sample Receipt

Upon receipt by the laboratory, sample integrity will be inspected and documented on the COC or associated document (i.e., a sample receipt report or similar document). Information to be noted on the COC includes: name of person inspecting cooler, integrity of custody seals, sample cooler temperature, evidence of preservation, physical condition of sample container, and airbill number. The COCs will be reviewed for completeness. If any sample integrity or sample ID problems or discrepancies are found, the Field Supervisor or Project Manager will be notified immediately. A COC addendum or sample receipt report may be used to document the corrective actions used to address any COC discrepancies. If an addendum is not used, corrective actions used to correct COC discrepancies must be recorded directly on the COC. Samples will be stored in a specially designated area that is clean, dry, and refrigerated (if needed).

Sample Labeling

The field sample number will be recorded on the sample inventory, the COC, and on the sample label. All samples will be assigned discrete sample identification numbers (sample control numbers) upon receipt by the laboratory. The laboratory sample control number will remain the same throughout the analysis and data entry procedures. Final results will be reported with both the field sample ID and the laboratory sample control number.

Sample Custody

The laboratory will be responsible for maintaining an accurate custody record for each sample in the lab. Records will be maintained to document the date and time the sample is checked out of sample storage for analysis and the date and time at which the sample is returned. The Laboratory Project Manager or laboratory contact will be responsible for supplying the Field Supervisor (or their designee) with a sample acknowledgment form within 24 hours of sample

receipt. This form will provide sample receipt information, sample log-in information, and the laboratory project number for the samples. A completed, signed COC will be sent by the laboratory to the Project Manager with the final data report.

5.5 ANALYTICAL PROCEDURES

Analytical methods for investigation activities are presented in Section 4.6 of this Work Plan. The test methods selected as part of this investigation program are standard EPA or ASTM procedures.

Detailed laboratory QC requirements are contained within each individual method SOP. The minimum requirements for the QC samples are outlined below. Laboratory QC sample results are reported with the data report.

Laboratory Duplicates, Matrix Spikes, and Matrix Spike Duplicates

Duplicate analysis is performed as a measurement of precision on the analytical process. Laboratory duplicates are independently repeated measurements of the same sample, which are performed by the same analyst and under the same conditions. The sample is split in the laboratory and each fraction is carried through all stages of preparation and analysis. The RPD is calculated from the two sample results. The duplicate procedure is performed at least once per 20 samples for chemical analyses which do not include matrix spike/matrix spike duplicates (MS/MSDs).

MSs are prepared by adding a known amount of each target analyte (or a subset thereof) to a known amount of sample. The MS is added at the beginning of the procedure and is carried through the entire measurement process. The sample itself (without an MS) is also carried through the analytical process. In order to produce reliable recovery results, the spike level must be similar to the sample concentration. Because the MSs are prepared and analyzed at the same time as the sample, only a reasonable estimate of the spike level can be made. Where samples are collected in field areas that are expected to have high concentrations, they will be identified for the laboratory, and corresponding spike levels can be used. The amount of the spike should be at least four times the amount in the unspiked sample.

The spike recovery measures the effects of interferences caused by the sample matrix in the analytical process. The MS recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{spiked sample result} - \text{sample result}}{\text{theoretical spike concentration}} \times 100$$

For chemical analyses, the matrix spike procedure is performed once per batch of 20 samples. The MS is prepared and analyzed in duplicate and the second spike is called the MSD. This procedure evaluates the precision associated with the procedure and the analyst performing the procedure and is calculated as a RPD as described above.

If a site sample is to be used as an MS/MSD, the sample to be used shall be designated on the COC. The MS/MSD is used to document the bias of a method due to sample matrix, not to control the analytical process and thus laboratory corrective action is not instituted based on MS/MSD results.

Laboratory Control Standard (LCS) and Laboratory Control Standard Duplicates (LCSDs)

The laboratory control sample (LCS) is an aliquot of a solid or aqueous certified reference material containing a known amount of each target analyte being measured. The LCS is treated like a field sample from the beginning of the procedure and is carried through the entire measurement process. The amount of the spike should be at a level less than or equal to the midpoint of the calibration curve for each analyte. For chemical analyses, the LCS is analyzed once per batch of 20 samples.

The percent recovery of the target analytes in the LCS assists in determining whether the procedure is in control. It is further used to evaluate the accuracy and bias of all or a portion of the measurement process. If insufficient quantity of sample is provided to perform a matrix spike and matrix spike duplicate, a duplicate LCS (LCSD) is prepared and analyzed and the RPD is calculated as described previously.

Detectability Check Sample

For chemical analyses, the laboratory should routinely check the instrument MDL to verify the laboratory's ability to reliably detect the parameter at the MDL that is used for reporting detected

results and calculation of non-detected results. The detectability check standard should be routinely analyzed and the results maintained on file with the MDL data.

Method Blank

The method blank is analyte-free water or solid material that is processed simultaneously with and under the same conditions as the samples. For chemical analyses, the method blank is analyzed once per batch of 20 samples to demonstrate that the analytical system itself is not contaminated with the analyte(s) being measured. The method blank results should be below the Method Quantitation Limit or corrective action must be taken. No qualification is warranted if a sample result from the sample group is greater than or equal to five times the associated blank concentration. Analytical results less than five times the associated blank concentration are qualified as non-detected.

Negative Control

A control sediment is one that is essentially free of contaminants and is used routinely to assess the acceptability of a bioassay test; it is not necessarily collected near the site of concern. A control sediment provides a measure of test acceptability, evidence of test organism health, and a basis for interpreting data obtained from the test sediments. Any study in which organisms in the negative control do not meet performance criteria must be considered questionable. The negative control is included in each batch of bioassay test samples.

Positive Control (Reference Toxicant)

A reference-toxicity test is one conducted with reagent-grade reference chemical to assess the sensitivity of the bioassay test organisms response to a toxicant challenge. Deviations outside an established normal range ($\pm$2 SD, 95% confidence limits) may indicate a change in the sensitivity of the test organism population. Reference-toxicity tests are most often performed in the absence of sediment and are performed at least once every six months.

Additional Method Specific QC Requirements

Additional QC samples may be run (e.g., continuing calibration samples), as specified in the method SOPs. The requirements for these samples, their acceptance criteria, and corrective action are method-specific.

Failures in Quality Control and Corrective Action

All qualified data are evaluated by the Project Manager, in consultation with the QA Manager. Since the differences between field duplicate sample results are used to assess the entire sampling process, including environmental variability, the arbitrary rejection of results based on pre-determined limits is not practical. Therefore, the professional judgment of the Project Manager and QA Manager will be relied upon in evaluating results. Rejecting sample results based on wide variability is a possibility. Field blank values exceeding the acceptability criteria may automatically invalidate the sample, especially in cases where high blanks may be indicative of contamination that causes a result to exceed the standard. Field duplicate excursions will be noted. Equipment blank results are also scrutinized very closely. Corrective action will involve identification of the cause of the failure where possible. Response actions may include re-analysis of questionable samples. In some cases, a site may have to be re-sampled to achieve project goals.

Laboratory measurement quality control failures are evaluated by the Laboratory Project Manager and findings reported to the Project Manager.

Standards Traceability

All standards used in the laboratory are traceable to certified reference materials. Standards preparation is fully documented and maintained in a standards log book. Each document includes information concerning the standard identification, starting materials, including concentration, amount used and lot number, date prepared, expiration date and preparer's initials or signature. The reagent bottle is labeled in a way that traces the reagent back to the preparation.

Failures in Measurement Systems and Corrective Actions

In many cases, the field technician or lab analyst will be able to correct problems. If the problem is resolved by the field technician or lab analyst, he/she will document the problem on the field data sheet or laboratory record and complete the analysis. If the problem is not resolvable, then it is conveyed to the Laboratory Project Manager, who will make the determination and notify the QA Manager. If the analytical system failures may compromise the sample results, the resulting

data will not be reported. The nature and disposition of the problem is reported on the data report, which is sent to the Project Manager.

5.6 PREVENTIVE MAINTENANCE

5.6.1 Field Instrument Preventive Maintenance

Field instruments are checked and calibrated prior to beginning the field program and daily before use to verify that instruments are in good working order. Routine preventive maintenance procedures are specified in the relevant operation manuals. Additional details on the field equipment to be used in this project are provided in applicable procedures specified in the Field Sampling Plan.

5.6.2 Laboratory Instrument Routine Maintenance Activities

As part of the laboratory QA/QC program, a routine preventive maintenance program will be conducted by the laboratories to minimize the occurrence of instrument failure or other system malfunction. The laboratory workload will be scheduled to accommodate planned downtime required to complete routine maintenance procedures. Trained operators will complete routine maintenance procedures (e.g., changing oven fans, replacing electronic control boards, changing vacuum pump oil, cleaning, etc.) for GC/MS instruments. An inventory of spare parts will be maintained to facilitate timely repair of instruments and minimize downtime.

Records of preventive maintenance activities for each piece of equipment will be maintained in Calibration and Maintenance log books assigned to that instrument. Preventive maintenance performed during the project will be noted in the field logbook and the instrument Calibration and Maintenance log book.

5.6.3 Inspection/Acceptance Requirements for Supplies and Consumables

Supplies and spare parts should be maintained for both field and laboratory instruments to assure timely completion of sample screening and analysis. For field work, critical spare parts such as batteries will be kept on-site to reduce downtime. Backup instruments and equipment should be available on-site or within 1 day shipment to avoid delays in the field schedule.

5.7 DATA MANAGEMENT AND REPORTING

Data management provides a process for tracing the path of the data from their generation in the field or laboratory to their final use or storage. The following elements are included in this process: recording, validation, transformation, transmittal, reduction, analysis, tracking, and storage and retrieval.

Data Recording

Sample collection will be documented and tracked using field log forms, field logbook entries, and Chain-of-Custody Records. Field personnel will complete these forms, which then will be reviewed for correctness and completeness by the Field Supervisor. Copies of these forms will be maintained in the project files.

Data Transformation

Since data will be collected and/or reported using proper units according to this QAPP, no data transformation is expected. If data transformation is necessary, the transformation procedures will be added to this QAPP.

Data Transmittal

The Field Supervisor will be responsible for assuring that field data are entered onto the appropriate field data forms, and will report any problems to the Project Manager. Field Supervisors will submit the complete field data forms to the Project Manager for review and error checking.

Field Supervisors will also ensure that all samples collected in the field are submitted to the laboratory according to the methods outlined in this QAPP or the FSP. The laboratory will submit to the Project Manager or Field Supervisor the analytical data results in their standard hard-copy format (including raw data format) and in an electronic data deliverable (EDD) format prior to sending the final data report in PDF to the Project Manager. The EDD shall be in space or comma-delimitated ASCII format or in Excel spreadsheet format that will allow for easy integration into a digital database.

Once reviewed by the Project Manager or Field Supervisor for obvious transcription or reporting errors, the final data report in both hard-copy and EDD formats will be transmitted and ready for validation by the QA Manager. Following data validation, any data qualifiers added to data during the validation process will be imported into the project database. Entry or upload of EDDs and data qualifiers into the project database will be completed by a designee of the Project Manager. The data and qualifiers will be initially verified by the individual entering the data. Upon completion of the initial verification step, a report will be generated of the data and verified by the Project Manager against the original data. Only final versions of electronic data will be entered into the database. All electronic data will be verified before and after incorporation into the database against the hard copy reports that accompany the data.

All qualified data will be included with the data packages during all subsequent data transmittal processes. The final hard copy data validation checklists will be included with the data in the final BERA report document.

All field forms and lab data will be organized and stored by sample location allowing for easy access if needed. Data can be transferred electronically either on disc, CD, tape or as an email attachment.

Data Storage and Retrieval

PBW's Project Manager is responsible for project data storage and retrieval. Laboratory data that are stored electronically will be archived electronically, and where printed as part of the paper data report package, will also be archived in paper form. Both the electronic data and hard copies will be maintained in PBW's Round Rock, TX office. In general, all records and data must be retained for a period of 10 years following commencement of construction or of any remedial action which is selected following completion of the RI/FS, per Section XX, Paragraph 79 of the UAO.

5.7.1 Data Review: Verification, Validation, and Integrity

For the purpose of this document, verification means the processes taken to determine compliance of data with project requirements, including documentation and technical criteria. Validation means those processes taken independently of the data-generation processes to determine the

usability of data for its intended use(s). Integrity means the processes taken to assure that no falsified data will be reported.

All data obtained from field and laboratory measurements will be reviewed and verified for conformance to project requirements, and then validated against the project objectives. Data supported by appropriate quality control results that meet the project objectives defined for this project will be considered acceptable without qualification. Data associated with quality control results that do not meet the project objectives defined for this project will be assigned appropriate qualifiers reflecting the potential impact on data usability. Analytical data will be considered usable unless rejected during the validation process.

The Field Supervisor is responsible for ensuring that field data are properly reviewed and verified for integrity by reviewing field equipment calibration records and verifying proper field procedures. The Analytical Lab Project Manager is responsible for ensuring that laboratory data are scientifically valid, defensible, of acceptable precision and accuracy, and reviewed for integrity and indicates this by signing the data package Narrative. The QA Manager will be responsible for ensuring that all laboratory data are properly reviewed and verified, and submitted in the required format to the project database. The QA Manager is responsible for validating the laboratory data and documenting the review. Finally, the Project Manager, with the concurrence of the QA Manager, is responsible for verifying that all data to be reported meet the objectives of the project and are suitable for reporting.

Verification and Validation Methods

All data will be verified to ensure they are representative of the samples analyzed and locations where measurements were made, and that the sample results and associated quality control data conform to project specifications. The staff and management of the respective field, laboratory, and data management tasks are responsible for the integrity, validation and verification of the data each task generates or handles throughout each process. The field and laboratory tasks ensure the verification of raw data, electronically generated data, and information on COC forms and hard copy output from instruments. The Analytical Lab Project Manager will document the review of the reported data per the laboratory's QA Plan.

Verification, validation and integrity review of all laboratory data will be performed or supervised by the QA Manager. The data to be verified are evaluated against project specifications (and are checked for errors, especially errors in transcription, calculations, and data input. The QA Manager will validate all reported laboratory data in accordance with the project Data Validation Standard Operating Procedure found in Appendix F of the RI/FS QAPP (PBW, 2006c). All laboratory data will be validated using a Level III data review. For critical samples, a Level IV review may be instituted. The validation will be documented on the Validation Checklist included in the SOPs and data qualifiers will be added to the database as appropriate. The SOPs include guidelines for applying data qualifiers. Generally, data will be rejected for use if the holding time is grossly exceeded or the QC data indicates an extremely low bias (<10% true value) in the measurement.

Potential outliers are identified by the QA Manager and Project Manager by examining results for unreasonable data, or identified using computer-based statistical software. If a question arises or an error or potential outlier is identified, the Field Supervisor or the Analytical Lab Project Manager responsible for generating the data is contacted to resolve the issue. Issues that can be corrected are corrected and documented electronically or by initialing and dating the associated paperwork. If an issue cannot be corrected, the QA Manager and/or the Project Manager will determine the appropriate course of action, or the data associated with the issue are rejected.

The Project Manager and QA Manager are each responsible for validating that the verified data are scientifically valid, defensible, of known precision, accuracy, integrity, meet the project objectives of the project, and are reportable. One element of the validation process involves evaluating the data again for anomalies. The QA Manager or Project Manager may designate other experts familiar with the project to perform this evaluation. Any suspected errors or anomalous data must be addressed by the manager of the task associated with the data before data validation can be completed.

5.8 SYSTEMS AND PERFORMANCE AUDITS

Performance and system audits may be conducted to verify that sampling and analysis are performed in accordance with applicable SOPs specified for field and laboratory activities. The audits of field and laboratory activities include two independent components: internal and external audits.

5.8.1 Field Performance and System Audits

Internal Field Audits

Internal audits of field activities, including sampling and field measurements, will be conducted by the BERA Investigation Manager or a designated alternate. Additional team members may also be present during various phases of the audits. These audits will be conducted to evaluate performance, verify that procedures are followed, and correct deficiencies in the execution of field procedures.

An internal field audit will be conducted at least once at the beginning of the site sample collection activities to verify that established procedures are being followed.

To verify compliance with established procedures and implementation of appropriate QA procedures, internal audits will involve the review and examination of the following: i) field measurement and sampling records, ii) instrument operation and calibration records, iii) sample collection documentation, iv) sample handling and packaging procedures, and v) chain-of-custody procedures. Results of field performance audits will be documented on a field audit checklist. If the first audit reveals significant deficiencies, one or more follow-up audits will be conducted to verify that QA procedures are maintained throughout the remainder of the investigation.

5.8.2 Laboratory Performance and System Audits

Internal Laboratory Audits

Internal system and performance audits at the analytical laboratory will be the responsibility of the Laboratory QA Manager. The internal laboratory system audit will be conducted on an annual basis, and the internal lab performance audit on a quarterly basis. Performance and systems audits for sampling and analysis operations will include on-site review of laboratory quality assurance systems and on-site review of equipment for calibration and measurement techniques.

External Laboratory Audits

One or more external laboratory audits may be conducted by the U.S. EPA Region 6 Project Coordinator. External laboratory audits will be conducted at the discretion of the U.S. EPA Region 6 Project Coordinator. External lab audits will include, but not be limited to, review of laboratory analytical procedures, laboratory on-site audits, and/or submission of performance evaluation samples to the laboratory for analysis.

5.9 CORRECTIVE ACTIONS

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or poor QC performance which can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation and data assessment. All proposed corrective actions should be documented as well as the steps taken to implement the corrective action. Corrective action should only be implemented after approval by the Project Manager or his designee. If immediate corrective action is required, approvals secured by telephone from the Project Manager should be documented.

For noncompliance problems, a formal corrective action program will be developed and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Project Manager. If the problem is related to an analytical procedure affecting the quality of data produced, this information will be promptly communicated to the Analytical Lab Project Manager, the Project Manager and the QA Manager. Implementation of corrective action will be confirmed in writing through the same channels.

Any nonconformance with the established QC procedures will be identified and corrected in accordance with this QAPP. The Project Manager, or his designee, will issue a nonconformance report for each nonconformance condition and include a copy of this report in the project's files.

5.9.1 Field Corrective Action

Corrective action in the field may be needed when the sample program is changed (i.e., more/less samples, sampling locations or frequencies other than those specified in the WP or FSP) or when sampling procedures and/or field procedures require modification due to unexpected conditions. In general, the field team may identify the need for corrective action. The field staff, in conjunction with the field team leader, will recommend a corrective action. The Project Manager will approve the corrective measure, which will be implemented by the field team. It will be the responsibility of the Project Manager to ensure the corrective action has been implemented.

If the corrective action will supplement the WP or FSP, using existing and approved procedures in the QAPP, corrective action approved by the Project Manager will be documented. If corrective actions result in less samples, alternate sampling locations, etc., which may cause project QA objectives not to be achieved, it will be necessary that all levels of project management concur with the proposed action.

Corrective action resulting from internal field audits will be implemented immediately if data quality would be adversely affected due to unapproved or improper use of approved methods. The QA Manager will identify deficiencies and recommend corrective action to the Project Manager. Implementation of corrective actions will be performed by the field team under the direction of the Project Manager.

Corrective actions will be documented in the field notebook or field forms. No staff member will initiate corrective action without prior communication of findings through the proper channels. If the actions taken are insufficient to correct the problem identified, work may be stopped by the Project Manager. If at any time a corrective action issue is identified which directly impacts the project objectives, the Project Coordinator will be notified immediately.

5.9.2 Laboratory Corrective Action

Corrective actions in the laboratory may occur prior to, during or after initial analyses. As such, the initial analyses must be performed quickly enough to allow time for reanalysis within the

required holding time. A number of conditions, such as broken sample containers, may be identified during sample login or just prior to analysis. The Analytical Laboratory Project Manager will notify the QA Manager of such conditions prior to analysis. Following consultation with lab analysts and section leaders, it may be necessary for the Analytical Laboratory Project Manager to approve the implementation of corrective action. Some conditions that may trigger corrective action or optional procedures during or after analysis include dilution of samples, sample reanalysis when certain quality control criteria are not met, etc.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the control limits for precision or accuracy;
- Sample results are outside the instrument calibration range;
- Laboratory method blanks contain target analytes above acceptable levels;
- Deficiencies are detected during internal or external audits or from the results of performance evaluation samples; or
- Inquiries concerning data quality are received.

The following specific instances require laboratory corrective action:

- The laboratory method blanks contain target analytes above the MQL and any associated sample contains the analyte at a concentration less than five times that in the blank.
- The LCS recovery is less than 10% for any organic target analyte or 30% for any inorganic analyte.
- The LCS recovery is outside the control limit for more than 1/2 of the target analytes for multi-analyte analyses such as PAHs.
- The surrogate recovery is less than 10% for any single surrogate.
- The MS recovery is less than 30% for any inorganic analyte.
- The internal standard area is less than 25% (i.e., -75%) of that in the midpoint standard for any single internal standard.

The corrective action shall include reanalyzing (and extracting or digesting, as applicable) the affected samples and/or immediate notification of the QA Manager.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the analytical procedures for possible errors, checks the instrument calibrations and performance, etc. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor or Analytical Laboratory Project Manager for further investigation. Once resolved, full documentation of the corrective action procedure is filed. These corrective actions are performed prior to release of the data from the laboratory. All corrective actions associated with sample analyses for this project will be documented and reported in the sample package narrative.

5.9.3 Corrective Action During Data Validation and Data Assessment

The need for corrective action may be identified during either data validation or data assessment. Potential types of corrective action may include re-sampling, reanalysis of samples, or reprocessing of the sample data. These actions are dependent upon the ability to mobilize the field team and whether the data to be collected are necessary to meet the required QA objectives. If the QA Manager identifies a corrective action situation, it is the Project Manager who will be responsible for approving the implementation of corrective action. All corrective actions of this type will be documented by the QA Manager.

5.10 QUALITY CONTROL REPORTS

5.10.1 Laboratory Data Report

Laboratory data reports contain the results of all specified QC measures identified in Section 5.5, including but not limited to equipment blank, filter and reagent blanks, field blanks, laboratory duplicates, laboratory control standards, calibration, and matrix spikes. For chemical analyses, this is generally considered a Level III data report (see section 2.7.4 of RI/FS QAPP). This information is reviewed by the QA Manager and compared to the pre-specified acceptance criteria to determine acceptability of the data before forwarding to the Project Manager.

5.10.2 Reports to Project Management

The Field Supervisor will report to the Project Manager daily following each field monitoring event. A brief written report will be sent via e-mail to the Project Manager that documents any problems, delays, or corrective actions that may be required or that may affect the subsequent

sampling efforts. The report will also include a brief synopsis of the work conducted during the field monitoring event.

5.11 DECONTAMINATION PROCEDURES

Site personnel will perform decontamination in accordance with PBW SOP No.13: Equipment Decontamination, and the applicable SOPs for sampling sediments (RI/FS Field Sampling Plan, PBW, 2006b). Following sediment sample collection, the empty sampler should be rinsed and decontaminated using water and an Alconox® or an equivalent detergent, and rinsed with deionized water. The sampler and associated equipment is decontaminated before use and between sample sites. In addition, the sampler will be rinsed with Site water before samples are collected. Equipment used for sample collection, sub-sampling, and sample mixing will be stainless steel or Teflon®.

5.12 MANAGEMENT OF INVESTIGATION DERIVED WASTES

Due to the nature of the investigation, investigation derived wastes are not expected to be produced. If any wastes are generated they will be managed in accordance with the procedures described in the RI/FS FSP (PBW, 2006b) (Section 7.0).

6.0 HEALTH AND SAFETY PROCEDURES

The overall health and safety objective is to perform the field tasks in a manner that minimizes the potential for accidents or injuries, and minimizes the potential for worker exposure to hazardous chemicals. Details of the health and safety procedures are provided in the Site-Specific Health and Safety Plan (HSP) (PBW, 2005), dated August 17, 2005.

The HSP applies to the field activities described in this FSP that will be performed during the RI/FS at the Site. The HSP was prepared to comply with the requirements of 29 CFR 1910.120 (b)(4). The primary purpose of the plan is to provide the results of a hazard assessment conducted for the prescribed work tasks, and the health and safety requirements and protocols that will minimize hazards to site workers.

A copy of the HSP will be kept on site at all times during field activities. All personnel will complete the Safety Compliance Agreement provided in Appendix A of the HSP. Other health and safety documentation are detailed in the HSP.

7.0 REFERENCES

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Tables

TABLE 1
ASSESSMENT ENDPOINTS AND MEASURES

Guild	Receptor of Potential Concern	Assessment Endpoint for BERA	Ecological Risk Questions	Testable Hypotheses	Measures of Effects	Measures of Exposure	Measures of Ecosystem and Receptor Characteristics	Toxicity Testing
Invertebrates	Earthworm	Protection of soil invertebrate community from uptake and direct toxic effects on detritivore abundance, diversity, productivity from COPECs in soil.	Does exposure to COPECs in soil adversely affect the abundance, diversity, productivity, and function?	Concentrations of COPECs in soil are adversely affecting invertebrate receptors.	Invertebrate receptor response to identified COPECs in North Area soil.	4,4'-DDT, Aroclor-1254, barium, chromium, copper and zinc concentrations in soils. Sample locations based on gradient of COPEC concentrations.	Invertebrate receptor feeding behavior, growth and reproduction.	Earthworm *(Eisenia fetida)* (28 day chronic survival and growth)
Benthos and zooplankton	Polychaetes	Protection of benthic and water-column invertebrate communities from uptake and direct toxic effects on abundance, diversity, and productivity from COPECs in sediment and surface water.	Does exposure to COPECs in sediment and surface water adversely affect the abundance, diversity, productivity, and function?	Concetrations of COPECs in sediment and/or surface water are adversely affecting benthic receptors.	Benthic receptor response to identified COPECs in Intracoastal Waterway sediments and wetland sediments/surface water. Locations chosen on a gradient of COPEC concentrations.	COPEC concentrations in Intracoastal Waterway and wetland sediments and surface water. Sample locations for sediments based on gradient of COPEC concentrations.	Benthic receptor feeding behavior, growth and reproduction.	*Leptocheirus plumulosus* (28d chronic; survival, growth, reproduction); *Neanthes arenaceodentata (28d chronic; survival, growth); Mysidopsis bahia (7d chronic; survival, growth)*
Vertebrate Fish	Fish Community	Protection of fish communities from uptake and direct toxic effects on abundance, diversity, and productivity from COPECs in sediment and surface water.	Does exposure to COPECs in surface water adversely affect the abundance, diversity, productivity, and function?	Concetrations of COPECs in surface water are adversely affecting fish communities.	Fish Communities response to identified COPECs in wetland and pond surface water in the vicinity of concentrations exceeding applicable surface water benchmarks.	COPEC concentrations in wetland and pond surface water in the vicinity of sample locations relative to appropriate effect levels.	Fish community diversity and stability.	Not Applicable (see Section 3.4.1)

TABLE 2
SUMMARY OF SAMPLE LOCATIONS AND ANALYSES

Sample IDs, Location and Analytes	Selection Rationale		Sample Media	Analytical Methods and Organisms
North Area Soils				
BERA Sample ID: NAS01 North Soil Area RI/FS Sample ID:SB202 (mg/kg)	Location represents the high concentrations of the metals. Note that 4,4-DDT and Aroclor-1254 are below detection limits and not expected to be present.		Soil 0-0.5 feet below ground surface (ft bgs)	Metals US EPA 6010/6020 Total Organic Carbon ***Bioassay:*** Earthworm-28 day Chronic
4,4-DDT	0.00282 U			
Aroclor-1254	0.013 U			
Barium	476	H		
Chromium	128	H		
Copper	200	H		
Zinc	5640	H		
BERA Sample ID: NAS02 North Soil Area RI/FS Sample ID:SB204 (mg/kg)	Location represents the high concentration of 4,4'-DDT and Aroclor-1254, mid concentrations of chromium, copper, and zinc and a low concentration of barium.		Soil 0-2 ft bgs	Metals US EPA 6010/6020, PCBs US EPA Method 8082 Organochlorine Pesticides US EPA Method 8081 Total Organic Carbon ***Bioassay:*** Earthworm-28 day Chronic
4,4-DDT	0.395	H		
Aroclor-1254	6.35	H		
Barium	67.7	L		
Chromium	22.8	M		
Copper	92.3	M		
Zinc	134	M		
BERA Sample ID: NAS03 North Soil Area RI/FS Sample ID:SB206 (mg/kg)	Location represents the high concentration of barium, mid concentrations of chromium, copper, and zinc and the low concentration of 4,4'-DDT. Note that Aroclor-1254 is below detection limits and not expected to be present.		Soil 0-0.5 ft bgs	Organochlorine Pesticides US EPA Method 8081 Metals US. EPA 6010/6020 Total Organic Carbon ***Bioassay:*** Earthworm-28 day Chronic
4,4-DDT	0.00445	L		
Aroclor-1254	0.011 U			
Barium	426	H		
Chromium	23.1	M		
Copper	30.7	M		
Zinc	398	M		
BERA Sample ID: NAS04 North Soil Area RI/FS Sample ID:NE4SB11 (mg/kg)	Location represents the mid concentration of barium, copper,B1 and zinc and the low concentrations of chromium and Aroclor-1254. Note that 4,4'-DDT is below detection limits and not expected to be present.		Soil 0-0.5 ft bgs	Metals US EPA 6010/6020, PCBs US EPA Method 8082 Total Organic Carbon ***Bioassay:*** Earthworm-28 day Chronic
4,4-DDT	0.000148 U			
Aroclor-1254	0.0122	L		
Barium	153	M		
Chromium	11.5	L		
Copper	27.4	M		
Zinc	107	M		
BERA Sample ID: NAS05 North Soil Area RI/FS Sample ID:NE3SB09 (mg/kg)	Location represents the mid concentrations of the four metals and the low concentration of 4,4'-DDT. Note that Aroclor-1254 is below detection limit and not expected to be present.		Soil 0-0.5 ft bgs	Organochlorine Pesticides US EPA Method 8081 Metals US. EPA 6010/6020 Total Organic Carbon ***Bioassay:*** Earthworm-28 day Chronic
4,4-DDT	0.0108	L		
Aroclor-1254	0.00801 U			
Barium	145	M		
Chromium	30	M		
Copper	27.8	M		
Zinc	288	M		
BERA Sample ID: NAS06 North Soil Area RI/FS Sample ID:NE3SB09 (mg/kg)	Location represents low concentrations of the four metals. Note that Aroclor-1254 and 4,4-DDT are below detection limits and not expected to be present.		Soil 0-0.5 ft bgs	Metals US. EPA 6010/6020 Total Organic Carbon ***Bioassay:*** Earthworm-28 day Chronic
4,4-DDT	0.00016 U			
Aroclor-1254	0.00415 U			
Barium	46.1	L		
Chromium	11.7	L		
Copper	8.04	L		
Zinc	32.6	L		
BERA Sample ID: NAS07 North area Background Soil Location Background Soil BSS-01 (mg/kg)	Represents background location with high zinc concentration		Soil 0-0.5 ft bgs	Organochlorine Pesticides US EPA Method 8081 Metals US EPA 6010/6020, PCBs US EPA Method 8082 Total Organic Carbon ***Bioassay:*** Earthworm-28 day Chronic
Chromium	17.6			
Zinc	969			

TABLE 2
SUMMARY OF SAMPLE LOCATIONS AND ANALYSES

Sample IDs, Location and Analytes	Selection Rationale	Sample Media	Analytical Methods and Organisms
BERA Sample ID: NAS08 North area Background Soil Location Background Soil BSS-02	Represents background location with low zinc concentration	Soil 0-0.5 ft bgs	Organochlorine Pesticides US EPA Method 8081 Metals US EPA 6010/6020, PCBs US EPA Method 8082 Total Organic Carbon
			Bioassay:
Barium	361		Earthworm-28 day Chronic
Chromium	17.6		
Zinc	81.2		
BERA Sample ID: NAS09 North area Background Soil Location Background Soil BSS-03	Represents background location with low zinc concentration	Soil 0-0.5 ft bgs	Organochlorine Pesticides US EPA Method 8081 Metals US EPA 6010/6020, PCBs US EPA Method 8082 Total Organic Carbon
			Bioassay:
Chromium	20.1		Earthworm-28 day Chronic
Zinc	77		

TABLE 2
SUMMARY OF SAMPLE LOCATIONS AND ANALYSES

Sample IDs, Location and Analytes	Selection Rationale		Sample Media	Analytical Methods and Organisms
Intracoastal Waterway Sediment (All Locations 0-0.5 ft bgs)				
BERA Sample ID: EIWSED01 Intracoastal Waterway Sediment RI/FS Sample ID: IWSE-01 (mg/kg)	Location represents the high concentration of 4,4-DDT and low concentrations of four PAHs. Note that hexachlorobenzene is below detection limit and not expected to be present.		Sediment	PAHs US EPA Method 8270 Organochlorine Pesticides US EPA Method 8081 Total Organic Carbon ***Bioassay:*** Amphipod - 28d Chronic, *Leptocheirus plumulosus* Polychaete - 28d Chronic, *Neanthes arenaceodentata*
4,4-DDT	0.00332	H		
Acenapthene	0.013 U			
Benzo(a)anthracene	0.0133 U			
Chrysene	0.0145	L		
Dibenz(a,h)anthracene	0.0126 U			
Fluoranthene	0.0309	L		
Fluorene	0.0129 U			
Hexachlorobenzene	0.0161 U			
Phenanthrene	0.0373	L		
Pyrene	0.0244	L		
			Pore Water	PAHs US EPA Method 8270 Organochlorine Pesticides US EPA Method 8081
BERA Sample ID EIWSED02 Intracoastal Waterway Sediment RI/FS sample ID: IWSE03 (mg/kg)	Location represents the high concentration of 6 PAHs, the mid concentration of two other PAHs and the low concentration of 4,4-DDT. Note that hexachlorobenzene is below detection limit and not expected to be present.		Sediment	PAHs US EPA Method 8270 Organochlorine Pesticides US EPA Method 8081 Total Organic Carbon ***Bioassay:*** Amphipod - 28d Chronic, *Leptocheirus plumulosus* Polychaete - 28d Chronic, *Neanthes arenaceodentata*
4,4-DDT	0.000575	L		
Acenapthene	0.0631	H		
Benzo(a)anthracene	0.395	H		
Chrysene	0.475	H		
Dibenz(a,h)anthracene	0.151	M		
Fluoranthene	0.804	H		
Fluorene	0.0406	H		
Hexachlorobenzene	0.0156 U			
Phenanthrene	0.508	M		
Pyrene	0.862	H		
			Pore Water	PAHs US EPA Method 8270 Organochlorine Pesticides US EPA Method 8081
BERA Sample ID: EIWSED03 Intracoastal Waterway Sediment RI/FS sample ID: IWSE04 (mg/kg)	Location represents the high concentration of 1 PAH, the mid concentration of chrysene, pyrene, fluoranthene, and 4,4'-DDT and the low concentration of dibenz(a,h)anthracene. Note that hexachlorobenzene is below detection limit and not expected to be present.		Sediment	PAHs US EPA Method 8270 Organochlorine Pesticides US EPA Method 8081 Total Organic Carbon ***Bioassay:*** Amphipod - 28d Chronic, *Leptocheirus plumulosus* Polychaete - 28d Chronic, *Neanthes arenaceodentata*
4,4-DDT	0.0011	M		
Acenapthene	0.0176 U			
Benzo(a)anthracene	0.018 U			
Chrysene	0.164	M		
Dibenz(a,h)anthracene	0.0694	L		
Fluoranthene	0.231	M		
Fluorene	0.0173 U			
Hexachlorobenzene	0.0217 U			
Phenanthrene	0.125	H		
Pyrene	0.285	M		
			Pore Water	PAHs US EPA Method 8270 Organochlorine Pesticides US EPA Method 8081
BERA Sample ID: EIWSED04 Intracoastal Waterway Sediment RI/FS sample ID: IWSE07 (mg/kg)	Location represents the high concentration of 1 PAH and hexachlorobenzene, the mid concentration of four PAHs and the low concentration of acenaphthene and phenanthrene.		Sediment	PAHs & Hexachlorobenzene US EPA Method 8270 Total Organic Carbon ***Bioassay:*** Amphipod - 28d Chronic, *Leptocheirus plumulosus* Polychaete - 28d Chronic, *Neanthes arenaceodentata*
4,4-DDT	0.000216 U			
Acenapthene	0.0239	L		
Benzo(a)anthracene	0.172	M		
Chrysene	0.197	M		
Dibenz(a,h)anthracene	0.235	H		
Fluoranthene	0.124	M		
Fluorene	0.0277	M		
Hexachlorobenzene	0.0319	H		
Phenanthrene	0.0645	L		
Pyrene	0.134	M		
			Pore Water	PAHs & Hexachlorobenzene US EPA Method 8270

TABLE 2
SUMMARY OF SAMPLE LOCATIONS AND ANALYSES

Sample IDs, Location and Analytes	Selection Rationale		Sample Media	Analytical Methods and Organisms
BERA Sample ID: EIWSED05 Intracoastal Waterway Sediment RI/FS sample ID: IWSE08 (mg/kg)	Location represents the mid concentration of pyrene and fluoranthene and the low concentrations of three PAHs and 4,4-DDT. Note that hexachlorobenzene is below detection limit and not expected to be present.		Sediment	PAHs US EPA Method 8270 Organochlorine Pesticides US EPA Method 8081 Total Organic Carbon ***Bioassay:*** Amphipod - 28d Chronic, *Leptocheirus plumulosus* Polychaete - 28d Chronic, *Neanthes arenaceodentata*
4,4-DDT	0.000481	L		
Acenapthene	0.0155 U			
Benzo(a)anthracene	0.0675	L		
Chrysene	0.0717	L		
Dibenz(a,h)anthracene	0.0151 U			
Fluoranthene	0.158	M		
Fluorene	0.0153 U			
Hexachlorobenzene	0.0192 U			
Phenanthrene	0.0756	L		
Pyrene	0.158	M		
			Pore Water	PAHs US EPA Method 8270 Organochlorine Pesticides US EPA Method 8081
BERA Sample ID: EIWSED06 Intracoastal Waterway Reference Sediment Sample located in Intracoastal Waterway Background Area near RI Sample location IWSE22	No impacts above screening values were indicated in the vicinity of this location during RI sampling.		Sediment	Organochlorine Pesticides US EPA Method 8081 PAHs & Hexachlorobenzene US EPA Method 8270 Total Organic Carbon ***Bioassay:*** Amphipod - 28d Chronic, *Leptocheirus plumulosus* Polychaete - 28d Chronic, *Neanthes arenaceodentata*
			Pore Water	PAHs & Hexachlorobenzene US EPA Method 8270 Organochlorine Pesticides US EPA Method 8081
BERA Sample ID: EIWSED07 Intracoastal Waterway Reference Sediment Sample located in Intracoastal Waterway Background Area near RI Sample location IWSE24	No impacts above screening values were indicated in the vicinity of this location during RI sampling.		Sediment	Organochlorine Pesticides US EPA Method 8081 PAHs & Hexachlorobenzene US EPA Method 8270 Total Organic Carbon ***Bioassay:*** Amphipod - 28d Chronic, *Leptocheirus plumulosus* Polychaete - 28d Chronic, *Neanthes arenaceodentata*
			Pore Water	PAHs & Hexachlorobenzene US EPA Method 8270 Organochlorine Pesticides US EPA Method 8081

TABLE 2
SUMMARY OF SAMPLE LOCATIONS AND ANALYSES

Sample IDs, Location and Analytes	Selection Rationale		Sample Media	Analytical Methods and Organisms
Wetland Sediment (All Locations 0-0.5 ft bgs)				
BERA Sample ID: EWSED01 Wetland Sediment RI/FS sample ID: 2WSED04-004 (mg/kg)	Location represents the high concentration of multiple COPECs including PAHs and pesticides and the low concentrations of copper, endrin aldehyde, lead and zinc. A mid concentration of nickel is also listed. Note that several COPECs are below detection limit and not expected to be present.		Sediment	PAHs US EPA Method 8270 Metals US EPA Method 6010/6020 Organochlorine Pesticides US EPA Method 8081 Total Organic Carbon Acid Volatile Sulfide/Simultaneously Extracted Metals Grain Size
2-Methylnaphthalene	0.153 U			
4,4-DDT	0.000939 U			***Bioassay:***
Acenaphthene	0.153 U			Amphipod - 28d Chronic, *Leptocheirus plumulosus*
Acenaphthylene	0.545	H		Polychaete - 28d Chronic, *Neanthes arenaceodentata*
Anthracene	0.334	H		
Arsenic	0.35 U			
Benzo(a)anthracene	0.126 U			
Benzo(a)pyrene	0.972	H		
Benzo(g,h,i)perylene	1.94	H		
Chrysene	4.05	H		
Copper	16	L		
Dibenz(a,h)anthracene	2.91	H		
Endrin Aldehyde	0.00431	L		
Endrin Ketone	0.013	H		
Fluoranthene	0.189 U			
Fluorene	0.12U			
gamma-chlordane	0.0036	H		
Indeno(1,2,3-cd)pyrene	1.94	H		
Lead	18.3	L		
Nickel	21.3	M		
Phenanthrene	0.111 U			
Pyrene	1.18	H		
Zinc	116	L		
			Pore Water	PAHs US EPA Method 8270 Metals US EPA Method 6010/6020 Organochlorine Pesticides US EPA Method 8081 Polychaete - 28d Chronic, *Neanthes arenaceodentata*
BERA Sample ID: EWSED02 Wetland Sediment RI/FS sample ID: 2WSED03-003 (mg/kg)	Location represents the high concentration of multiple COPECs including PAHs and pesticides and the low concentrations of copper, endrin ketone, lead and zinc. A mid concentration of several PAHs and nickel is also listed. Note that several COPECs are below detection limit and not expected to be present.		Sediment	PAHs US EPA Method 8270 Metals US EPA Method 6010/6020 Organochlorine Pesticides US EPA Method 8081 Total Organic Carbon Acid Volatile Sulfide/Simultaneously Extracted Metals Grain Size
2-Methylnaphthalene	0.173 U			
4,4-DDT	0.00107 U			***Bioassay:***
Acenaphthene	0.173 U			Amphipod - 28d Chronic, *Leptocheirus plumulosus*
Acenaphthylene	0.346	M		Polychaete - 28d Chronic, *Neanthes arenaceodentata*
Anthracene	0.241	M		
Arsenic	0.4 U			
Benzo(a)anthracene	U			
Benzo(a)pyrene	0.631	M		
Benzo(g,h,i)perylene	1.52	H		
Chrysene	2.73	M		
Copper	12.6	L		
Dibenz(a,h)anthracene	2.83	H		
Endrin Aldehyde	0.01	H		
Endrin Ketone	0.00619	L		
Fluoranthene	0.213 U			
Fluorene	0.135 U			
gamma-chlordane	0.000862 U			
Indeno(1,2,3-cd)pyrene	1.59	H		
Lead	17.2	L		
Nickel	20.9	M		
Phenanthrene	0.125 U			
Pyrene	0.729	M		
Zinc	115	L		
			Pore Water	PAHs US EPA Method 8270 Metals US EPA Method 6010/6020 Organochlorine Pesticides US EPA Method 8081 Polychaete - 28d Chronic, *Neanthes arenaceodentata* Endrin aldehyde, endrin ketone, gamma-Chlordane
BERA Sample ID: EWSED03 Wetland Sediment RI/FS sample ID: NF4SE13-013 (mg/kg)	Location represents the high concentration of arsenic, copper, nickel, and zinc, and low concentrations of PAHs; also, a mid concentration of 4,4-DDT, lead, and pyrene. Note that several COPECs are below detection limit and not expected to be present.		Sediment	PAHs US EPA Method 8270 Metals US EPA Method 6010/6020 Organochlorine Pesticides US EPA Method 8081 Total Organic Carbon Acid Volatile Sulfide/Simultaneously Extracted Metals Grain Size

TABLE 2
SUMMARY OF SAMPLE LOCATIONS AND ANALYSES

Sample IDs, Location and Analytes	Selection Rationale		Sample Media	Analytical Methods and Organisms
2-Methylnaphthalene	0.0122	L		
4,4-DDT	0.00254	M		***Bioassay:***
Acenaphthene	0.0103 U			Amphipod - 28d Chronic, *Leptocheirus plumulosus*
Acenaphthylene	0.0117 U			Polychaete - 28d Chronic, *Neanthes arenaceodentata*
Anthracene	0.0126	L		
Arsenic	12.8	H		
Benzo(a)anthracene	0.0106 U			
Benzo(a)pyrene	0.0105 U			
Benzo(g,h,i)perylene	0.133	L		
Chrysene	0.0904	L		
Copper	35.7	H		
Dibenz(a,h)anthracene	0.0555	L		
Endrin Aldehyde	0.000403 U			
Endrin Ketone	0.000505 U			
Fluoranthene	0.0117 U			
Fluorene	0.0102 U			
gamma-chlordane	0.000265 U			
Indeno(1,2,3-cd)pyrene	0.0951	L		
Lead	64.7	M		
Nickel	27.7	H		
Phenanthrene	0.0898	L		
Pyrene	0.109	M		
Zinc	903	H		
			Pore Water	PAHs US EPA Method 8270 Metals US EPA Method 6010/6020 Organochlorine Pesticides US EPA Method 8081 Polychaete - 28d Chronic, *Neanthes arenaceodentata*
BERA Sample ID: EWSED04 Wetland Sediment RI/FS sample ID: 2WSD17-17 (mg/kg)	Location represents the high concentration of several PAHs, arsenic, and lead, low concentrations of nickel. A mid concentration of several PAHs, copper, and zinc. Note that the organochlorine pesticides are below detection limit and not expected to be present.		Sediment	Metals US EPA Method 6010/6020 PAHs US EPA Method 8270 Total Organic Carbon Acid Volatile Sulfide/Simultaneously Extracted Metals Grain Size
2-Methylnaphthalene	0.053	H		
4,4-DDT	0.000829 U			***Bioassay:***
Acenaphthene	0.133	H		Amphipod - 28d Chronic, *Leptocheirus plumulosus*
Acenaphthylene	0.013 U			Polychaete - 28d Chronic, *Neanthes arenaceodentata*
Anthracene	0.257	M		
Arsenic	1.4	H		
Benzo(a)anthracene	0.724	M		
Benzo(a)pyrene	0.618	M		
Benzo(g,h,i)perylene	0.527	M		
Chrysene	0.743	M		
Copper	25.6	M		
Dibenz(a,h)anthracene	0.312	M		
Endrin Aldehyde	0.000706 U			
Endrin Ketone	0.000603 U			
Fluoranthene	1.43	M		
Fluorene	0.139	H		
gamma-chlordane	0.000669 U			
Indeno(1,2,3-cd)pyrene	0.752	M		
Lead	237	H		
Nickel	13.7	L		
Phenanthrene	1.18	H		
Pyrene	1.34	H		
Zinc	404	M		
			Pore Water	PAHs US EPA Method 8270 Metals US EPA Method 6010/6020 Polychaete - 28d Chronic, *Neanthes arenaceodentata*
BERA Sample ID: EWSED05 Wetland Sediment RI/FS sample ID: NB4SE08-008 (mg/kg)	Location represents the high concentration of several PAHs, 4,4-DDT, copper, and zinc, low concentrations of acenaphthylene, endrin aldehyde, and nickel. A mid concentration of several PAHs, arsenic, and lead. Note that two organochlorine pesticides are below detection limit and not expected to be present.		Sediment	Metals US EPA Method 6010/6020 PAHs US EPA Method 8270 Organochlorine pesticides US EPA Method 8081 Total Organic Carbon Acid Volatile Sulfide/Simultaneously Extracted Metals Grain Size
2-Methylnaphthalene	0.0396	M		
4,4-DDT	0.00922	H		***Bioassay:***
Acenaphthene	0.113	M		Amphipod - 28d Chronic, *Leptocheirus plumulosus*
Acenaphthylene	0.0291	L		Polychaete - 28d Chronic, *Neanthes arenaceodentata*
Anthracene	0.188	M		
Arsenic	3.53	M		
Benzo(a)anthracene	0.993	H		
Benzo(a)pyrene	1.3	H		
Benzo(g,h,i)perylene	0.862	M		
Chrysene	1.27	M		
Copper	39.6	H		
Dibenz(a,h)anthracene	0.337	M		
Endrin Aldehyde	0.00452	L		
Endrin Ketone	0.000458 U			
Fluoranthene	2.17	H		
Fluorene	0.127	H		

TABLE 2
SUMMARY OF SAMPLE LOCATIONS AND ANALYSES

Sample IDs, Location and Analytes	Selection Rationale		Sample Media	Analytical Methods and Organisms
gamma-chlordane	0.00024 U			
Indeno(1,2,3-cd)pyrene	1.1	H		
Lead	88.1	M		
Nickel	10.9	L		
Phenanthrene	1.3	H		
Pyrene	1.64	H		
Zinc	601	H		
			Pore Water	PAHs US EPA Method 8270 Metals US EPA Method 6010/6020
				Organochlorine pesticides US EPA Method 8081
				Polychaete - 28d Chronic, *Neanthes arenaceodentata*
BERA Sample ID: EWSED06 Wetland Sediment RI/FS sample ID: SPSE03 (mg/kg) (Location from Pond)	Location represents the high concentration of zinc, low concentrations of 4,4-DDT, chrysene, and pyrene. Mid concentration of arsenic copper, lead, nickel, and a PAH.		Sediment	Metals US EPA Method 6010/6020 PAHs US EPA Method 8270 Organochlorine pesticides US EPA Method 8081 Total Organic Carbon Acid Volatile Sulfide/Simultaneously Extracted Metals Grain Size
2-Methylnaphthalene	NA			
4,4-DDT	0.00157	L		***Bioassay:*** Amphipod - 28d Chronic, *Leptocheirus plumulosus* Polychaete - 28d Chronic, *Neanthes arenaceodentata*
Acenaphthene	NA			
Acenaphthylene	NA			
Anthracene	NA			
Arsenic	5.01	M		
Benzo(a)anthracene	NA			
Benzo(a)pyrene	NA			
Benzo(g,h,i)perylene	0.135	M		
Chrysene	0.0257	L		
Copper	26.8	M		
Dibenz(a,h)anthracene	NA			
Endrin Aldehyde	NA			
Endrin Ketone	NA			
Fluoranthene	NA			
Fluorene	NA			
gamma-chlordane	NA			
Indeno(1,2,3-cd)pyrene	NA			
Lead	30.5	M		
Nickel	20.6	M		
Phenanthrene	NA			
Pyrene	0.0265	L		
Zinc	999	H		
			Pore Water	PAHs US EPA Method 8270 Metals US EPA Method 6010/6020
				Organochlorine pesticides US EPA Method 8081
				Polychaete - 28d Chronic, *Neanthes arenaceodentata*
BERA Sample ID: EWSED07 Wetland Sediment RI/FS sample ID: 4WSED3 (mg/kg)	Location represents low to mid concentrations for the PAHs and metals. Organochlorine pesticides were not detected in this sample and are assumed not to be present.		Sediment	Metals US EPA Method 6010/6020 PAHs US EPA Method 8270 Total Organic Carbon Acid Volatile Sulfide/Simultaneously Extracted Metals Grain Size
2-Methylnaphthalene	0.00936 U			
4,4-DDT	0.00498 U			***Bioassay:*** Amphipod - 28d Chronic, *Leptocheirus plumulosus* Polychaete - 28d Chronic, *Neanthes arenaceodentata*
Acenaphthene	0.016	L		
Acenaphthylene	0.00746 U			
Anthracene	0.033	L		
Arsenic	0.12 U			
Benzo(a)anthracene	0.199	L		
Benzo(a)pyrene	0.227	L		
Benzo(g,h,i)perylene	0.209	M		
Chrysene	0.094	L		
Copper	27.6	M		
Dibenz(a,h)anthracene	0.00635 U			
Endrin Aldehyde	0.00579 U			
Endrin Ketone	0.00527 U			
Fluoranthene	0.176	L		
Fluorene	0.015	L		
gamma-chlordane	0.00423 U			
Indeno(1,2,3-cd)pyrene	0.408	M		
Lead	29.3	M		
Nickel	19.6	M		
Phenanthrene	0.135	M		
Pyrene	0.188	M		
Zinc	290	M		
			Pore Water	PAHs US EPA Method 8270 Metals US EPA Method 6010/6020
				Polychaete - 28d Chronic, *Neanthes arenaceodentata*

TABLE 2
SUMMARY OF SAMPLE LOCATIONS AND ANALYSES

Sample IDs, Location and Analytes	Selection Rationale	Sample Media	Analytical Methods and Organisms
BERA Sample ID: EWSED08 Wetland Sediment Reference Location near RI Sample Location 3WSED6	Location represents a reference/background location not impacted by site activities, but has similar physical attributes (e.g., grain size).	Sediment	Metals US EPA Method 6010/6020
			PAHs US EPA Method 8270
			Organochlorine pesticides US EPA Method 8081
			Total Organic Carbon
			Acid Volatile Sulfide/Simultaneously Extracted Metals Grain Size
			Bioassay: Amphipod - 28d Chronic, *Leptocheirus plumulosus* Polychaete - 28d Chronic, *Neanthes arenaceodentata*
		Pore Water	PAHs US EPA Method 8270 Metals US EPA Method 6010/6020
			Organochlorine pesticides US EPA Method 8081
			Polychaete - 28d Chronic, *Neanthes arenaceodentata*
BERA Sample ID: EWSED09 Wetland Sediment Reference Location near RI Sample Location 2WSED11	Location represents a reference/background location not impacted by site activities, but has similar physical attributes (e.g., grain size).	Sediment	Metals US EPA Method 6010/6020
			PAHs US EPA Method 8270
			Organochlorine pesticides US EPA Method 8081
			Total Organic Carbon
			Acid Volatile Sulfide/Simultaneously Extracted Metals Grain Size
			Bioassay: Amphipod - 28d Chronic, *Leptocheirus plumulosus* Polychaete - 28d Chronic, *Neanthes arenaceodentata*
		Pore Water	PAHs US EPA Method 8270 Metals US EPA Method 6010/6020
			Organochlorine pesticides US EPA Method 8081
			Polychaete - 28d Chronic, *Neanthes arenaceodentata*

TABLE 2
SUMMARY OF SAMPLE LOCATIONS AND ANALYSES

Sample IDs, Location and Analytes	Selection Rationale		Sample Media	Analytical Methods and Organisms
Surface Water				
EWSW01 Surface water location off-site north of the North Area near RI/FS sample location 2WSW1	Dissolved copper and total acrolein concentrations exceed ecological benchmarks for water		Surface Water	Metals US EPA 6010/6020 VOCs US EPA Method 8260 ***Bioassay*** 7d Chronic (growth and survival), *Mysidopsis bahia*
EWSW02 Surface water reference sample location off-site north of the North Area west of RI/FS surface water sample locations	No impacts above screening values were indicated in the vicinity of this location during RI sampling		Surface Water	Metals US EPA 6010/6020 VOCs US EPA Method 8260 ***Bioassay*** 7d Chronic (growth and survival), *Mysidopsis bahia*
EWSW03 Surface water location off-site north of the North Area near RI/FS sample location 2WSW6	Dissolved copper concentration exceeds ecological benchmark for water		Surface Water	Metals US EPA 6010/6020 VOCs US EPA Method 8260 ***Bioassay*** 7d Chronic (growth and survival), *Mysidopsis bahia*
EWSW04 Surface water from the pond area with silver concentrations greater than the benchmark (location not shown on Figure 9)	Dissolved silver concentration exceeds ecological benchmark for water		Surface Water	Metals US EPA 6010/6020 ***Bioassay*** 7d Chronic (growth and survival), *Mysidopsis bahia*

Notes:
1. Sample locations are provided on Figures 5 through 9.

H	represents a high concentration within the gradient
M	represents a mid concentration within the gradient
L	represents a low concentration within the gradient

NA - Not available.
U - Undetected.

TABLE 3
SUMMARY OF SAMPLE CONTAINERS, PRESERVATIVES, AND HOLD TIMES

Parameter	Sample Container and Preservative		Sample Storage	Maximum Holding Time
	Aqueous	Sediment		
Metals	250 ml glass or HDPE bottle, HNO3	4 oz glass or plastic	<6° C	6 months
PAHs	2x1000 ml amber glass	4 oz glass or plastic	<6° C	7 days water, 14 days soil (preparation); 40 days (analysis)
Organochlorine Pesticides	2x1000 ml amber glass	4 oz glass or plastic	<6° C	7 days water, 14 days soil (preparation); 40 days (analysis)
PCBs	2x1000 ml amber glass	4 oz glass or plastic	<6° C	7 days water, 14 days soil (preparation); 40 days (analysis)
Volatiles	3 x 40 ml VOA Vials, HCl	NA	<6° C	14 days
TOC	NA	250 ml plastic	<6° C	28 days
AVS/SEM	NA	100 grams glass or plastic	<6° C	14 days
Bioassay	1 gallon plastic	1L plastic	<6° C	8 weeks
Moisture	NA	4 oz glass jar	<6° C	NA

Notes:
1. NA = Not applicable to this analysis or matrix.
2. Sample volumes submitted for analysis of pore water may be reduced due to limited sample volume.

TABLE 4
ANALYTICAL METHODS

Media	COPECs	Test Method
Sediment		
Bulk Sediment	Toxicity (survival, growth, reproduction)	US EPA 600/R-01/020 28d chronic *Leptocheirus plumulosus*
Bulk Sediment	Toxicity (survival, growth)	ASTM E1611 28d chronic *Neanthes arenaceodentata*
Bulk Sediment	Metals	US EPA 6010B/6020
Bulk Sediment	Polynuclear Aromatic Hydrocarbons (PAHs) and hexachlorobenzene	US EPA 8270C
Bulk Sediment	Organochlorine Pesticides (4,4'-DDT, gamma chlordane, endrin aldehyde, endrin ketone)	US EPA 8081A
Bulk Sediment	Grain Size	ASTM D422
Bulk Sediment	Acid Volatile Sulfide/Simultaneously Extracted Metals (AVS/SEM)	US EPA Draft Analytical Method EPA/821/R-91/100
Bulk Sediment	Total Organic Carbon (TOC)	US EPA 9060
Aqueous		
Pore Water, Surface Water	Metals	US EPA 6010B/6020
Surface Water	Volatile Organic Compounds (Acrolein)	US. EPA 8260B
Pore Water	Polynuclear Aromatic Hydrocarbons (PAHs) and hexachlorobenzene	US EPA 8270C
Pore Water	Organochlorine Pesticides (4,4'-DDT, gamma-Chlordane, endrin aldehyde, endrin ketone)	US EPA 8081A
Surface Water	Toxicity (survival, growth)	US EPA 821/R-02/014 7d chronic *Mysidopsis bahia*
Soil		
Soil	Toxicity (survival, growth, reproduction)	Earthworm-28 day Chronic
Soil	Metals	US EPA 6010B/6020
Soil	Organochlorine Pesticides (4,4'-DDT, gamma chlordane, endrin aldehyde, endrin ketone)	US EPA 8081A
Soil	PCBs	US EPA 8082
Soil	Total Organic Carbon (TOC)	US EPA 9060

Notes:
1. Bioassay tests will be performed by PBS&J (Houston, Texas)
2. All other analyses will be performed by Columbia Analytical services (Kelso, Washington)
3. PAH compounds include acenaphthalene, acenaphthylene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(g,h,i)perylene, chrysene, dibenzo(a,h)anthracene, fluoranthene, flourene, indeno(1,2,3-cd)pyrene, phenathrene, and pyrene.

TABLE 5
PRECISION AND ACCURACY CRITERIA

Method	Prep Method	Matrix	Analyte	Surrogate (% Rec)	LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD)
Metals							
Sediment							
6020	3050B	Soil	Arsenic		78-122	70-130	30
6020	3050B	Soil	Copper		83-116	70-130	30
6020	3050B	Soil	Lead		79-121	70-130	30
6020	3050B	Soil	Nickel		81-118	70-130	30
6020	3050B	Soil	Zinc		73-121	70-130	30
Soil							
6020	3050B	Soil	Barium		81-119	70-130	30
6020	3050B	Soil	Chromium		80-119	70-130	30
6020	3050B	Soil	Copper		83-116	70-130	30
6020	3050B	Soil	Zinc		73-121	70-130	30
Water							
6020	Red. Precip.	Seawater	Copper		63-128	50-120	20
6020	Red. Precip.	Seawater	Nickel		88-112	60-126	20
6020	Red. Precip.	Seawater	Silver		80-110	67-103	20
6020	Red. Precip.	Seawater	Zinc		79-133	50-133	20
General Chemistry							
Sediment							
9060	NA	Soil	Total Organic Carbon		82-119	77-155	20
Pesticides							
Sediment							
8081A	3540C/3541	Soil	4,4'-DDT		46-151	19-154	40
8081A	3540C/3541	Soil	Endrin Aldehyde		32-132	10-129	40
8081A	3540C/3541	Soil	Endrin Ketone		47-135	19-139	40
8081A	3540C/3541	Soil	gamma-Chlordane		41-135	24-133	40
8081A	3540C/3541	Soil	Decachlorobiphenyl (Surr.)	15-130	NA	NA	NA
8081A	3540C/3541	Soil	Tetrachloro-*m*-xylene (Surr.)	21-112	NA	NA	NA
Water							
8081A	3520C/3535	Water	4,4'-DDT		42-143	28-139	30
8081A	3520C/3535	Water	Endrin Aldehyde		27-104	10-108	30
8081A	3520C/3535	Water	Endrin Ketone		30-124	34-113	30
8081A	3520C/3535	Water	gamma-Chlordane		47-113	35-119	30
8081A	3520C/3535	Water	Decachlorobiphenyl (Surr.)	35-128	NA	NA	NA
8081A	3520C/3535	Water	Tetrachloro-*m*-xylene (Surr.)	20-102	NA	NA	NA
Low-level SVOCs							
Sediment							
8270-SIM	3541	Soil	Acenaphthene		44-104	29-110	40
8270-SIM	3541	Soil	Acenaphthylene		41-110	32-106	40
8270-SIM	3541	Soil	Anthracene		47-112	31-115	40
8270-SIM	3541	Soil	Benz(a)anthracene		51-111	25-128	40
8270-SIM	3541	Soil	Benzo(a)pyrene		52-118	24-131	40
8270-SIM	3541	Soil	Benzo(g,h,i)perylene		46-114	24-127	40
8270-SIM	3541	Soil	Chrysene		54-111	25-132	40
8270-SIM	3541	Soil	Dibenz(a,h)anthracene		44-119	29-124	40

TABLE 5
PRECISION AND ACCURACY CRITERIA

Method	Prep Method	Matrix	Analyte	Surrogate (% Rec)	LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD)
8270-SIM	3541	Soil	Fluoranthene		51-111	22-138	40
8270-SIM	3541	Soil	Fluorene		49-105	29-117	40
8270-SIM	3541	Soil	Indeno(1,2,3-cd)pyrene		42-123	20-136	40
8270-SIM	3541	Soil	Phenanthrene		47-104	19-128	40
8270-SIM	3541	Soil	Pyrene		48-113	11-148	40
8270-SIM	3541	Soil	2,4,6-Tribromophenol (Surr.)	35-109	NA	NA	NA
8270-SIM	3541	Soil	Fluoranthene-d10 (Surr.)	27-106	NA	NA	NA
8270-SIM	3541	Soil	Fluorene-d10 (Surr.)	17-104	NA	NA	NA
8270-SIM	3541	Soil	Terphenyl-d14 (Surr.)	35-109	NA	NA	NA
Water							
8270-SIM	3520C	Water	Acenaphthene		44-113	45-114	30
8270-SIM	3520C	Water	Acenaphthylene		44-115	43-114	30
8270-SIM	3520C	Water	Anthracene		44-117	32-125	30
8270-SIM	3520C	Water	Benz(a)anthracene		48-125	41-128	30
8270-SIM	3520C	Water	Benzo(a)pyrene		43-134	35-132	30
8270-SIM	3520C	Water	Benzo(g,h,i)perylene		51-124	44-128	30
8270-SIM	3520C	Water	Chrysene		50-128	48-128	30
8270-SIM	3520C	Water	Dibenz(a,h)anthracene		49-133	43-135	30
8270-SIM	3520C	Water	Fluoranthene		48-128	48-134	30
8270-SIM	3520C	Water	Fluorene		48-118	45-123	30
8270-SIM	3520C	Water	Indeno(1,2,3-cd)pyrene		45-133	40-135	30
8270-SIM	3520C	Water	Phenanthrene		47-120	42-127	30
8270-SIM	3520C	Water	Pyrene		42-133	44-130	30
8270-SIM	3520C	Water	2,4,6-Tribromophenol (Surr.)	10-136	NA	NA	NA
8270-SIM	3520C	Water	Fluoranthene-d10 (Surr.)	31-105	NA	NA	NA
8270-SIM	3520C	Water	Fluorene-d10 (Surr.)	28-98	NA	NA	NA
8270-SIM	3520C	Water	Terphenyl-d14 (Surr.)	27-112	NA	NA	NA
SVOCs							
Sediment							
8270C-LL	3541	Soil-LL	Hexachlorobenzene		39-90	30-106	40
8270C-LL	3541	Soil-LL	2-Fluorobiphenyl (Surr.)	25-97	NA	NA	NA
8270C-LL	3541	Soil-LL	Nitrobenzene-d5 (Surr.)	27-91	NA	NA	NA
8270C-LL	3541	Soil-LL	Terphenyl-d14 (Surr.)	33-129	NA	NA	NA
Water							
8270C-LL	3520C	Water-LL	Hexachlorobenzene		42-102	31-101	30
8270C-LL	3520C	Water-LL	2-Fluorobiphenyl (Surr.)	31-94	NA	NA	NA
8270C-LL	3520C	Water-LL	Nitrobenzene-d5 (Surr.)	26-110	NA	NA	NA
8270C-LL	3520C	Water-LL	Terphenyl-d14 (Surr.)	40-127	NA	NA	NA
Volatiles							
Water							
8260B	5030B	Water	Acrolein		42-118	14-180	30
8260B	5030B	Water	1,2-Dichloroethane-D4 (Surr.)	59-127	NA	NA	NA
8260B	5030B	Water	4-Bromofluorobenzene (Surr.)	68-117	NA	NA	NA
8260B	5030B	Water	Dibromofluoromethane (Surr.)	73-122	NA	NA	NA
8260B	5030B	Water	Toluene-D8 (Surr.)	78-129	NA	NA	NA

TABLE 5
PRECISION AND ACCURACY CRITERIA

Method	Prep Method	Matrix	Analyte	Surrogate (% Rec)	LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD)
PCBs							
Soil							
8082	3540C/3541	Soil	Aroclor 1260	NA	53-129	20-185	40
8082	3540C/3541	Soil	Decachlorobiphenyl (Surr.)	35-133	NA	NA	NA
8082	3540C/3541	Soil	Tetrachloro-m-xylene (Surr.)	10-135	NA	NA	NA

Notes:
LCS - laboratory control samples
RPD - relative percent difference

TABLE 6
COMPARISON OF DETECTION LIMITS VS. ECOLOGICAL BENCHMARKS

Method	Analyte	Units	Benchmark	Method Detection Limit	Method Reporting Limit
Metals					
Sediment					
6020	Arsenic	mg/kg	8.2	0.06	0.5
6020	Copper	mg/kg	34	0.08	0.1
6020	Lead	mg/kg	46.7	0.009	0.05
6020	Nickel	mg/kg	20.9	0.00004	0.0002
6020	Zinc	mg/kg	150	0.2	0.5
Water					
6020	Copper	mg/L	0.0036	0.00003	0.0001
6020	Nickel	mg/L	0.0131	0.0002	0.0002
6020	Silver	mg/L	0.0002	0.008	0.02
6020	Zinc	mg/L	0.0842	0.00006	0.0005
Soil					
6020	Barium	mg/kg	330	0.005	0.5
6020	Chromium	mg/kg	0.4	0.03	0.2
6020	Copper	mg/kg	61	0.08	0.1
6020	Zinc	mg/kg	120	0.2	0.5
General Chemistry					
Sediment					
9060	Total Organic Carbon	mg/kg	--	0.02	0.05
Pesticides					
Sediment					
8081A	4,4'-DDT	mg/kg	0.00119	0.0002	0.001
8081A	Endrin Aldehyde	mg/kg	0.00267[C]	0.0002	0.001
8081A	Endrin Ketone	mg/kg	0.00267[C]	0.00006	0.001
8081A	gamma-Chlordane	mg/kg	0.00226[a]	0.00004	0.001
Water					
8081A	4,4'-DDT	mg/L	0.000001	0.000001	0.00001
8081A	Endrin Aldehyde	mg/L	0.000002[b]	0.000002	0.00001
8081A	Endrin Ketone	mg/L	0.000002[b]	0.000001	0.00001
8081A	gamma-Chlordane	mg/L	0.000004[a]	0.000001	0.00001
Low-level SVOCs					
Sediment					
8270-SIM	Acenaphthene	mg/kg	0.016	0.0003	0.005
8270-SIM	Acenaphthylene	mg/kg	0.044	0.0002	0.005
8270-SIM	Anthracene	mg/kg	0.0853	0.0002	0.005
8270-SIM	Benz(a)anthracene	mg/kg	0.261	0.0002	0.005
8270-SIM	Benzo(a)pyrene	mg/kg	0.43	0.0002	0.005
8270-SIM	Benzo(g,h,i)perylene	mg/kg	0.67[c]	0.0002	0.005
8270-SIM	Chrysene	mg/kg	0.384	0.0002	0.005
8270-SIM	Dibenz(a,h)anthracene	mg/kg	0.0634	0.0002	0.005
8270-SIM	Fluoranthene	mg/kg	0.6	0.0002	0.005
8270-SIM	Fluorene	mg/kg	0.019	0.0002	0.005
8270-SIM	Indeno(1,2,3-cd)pyrene	mg/kg	0.6[c]	0.0002	0.005
8270-SIM	Phenanthrene	mg/kg	0.24	0.0002	0.005
8270-SIM	Pyrene	mg/kg	0.665	0.0002	0.005
Water					
8270-SIM	Acenaphthene	mg/L	0.0404	0.000003	0.00002
8270-SIM	Acenaphthylene	mg/L	---	0.000002	0.00002
8270-SIM	Anthracene	mg/L	0.00018	0.000003	0.00002
8270-SIM	Benz(a)anthracene	mg/L	---	0.000003	0.00002

TABLE 6
COMPARISON OF DETECTION LIMITS VS. ECOLOGICAL BENCHMARKS

Method	Analyte	Units	Benchmark	Method Detection Limit	Method Reporting Limit
8270-SIM	Benzo(a)pyrene	mg/L	---	0.000002	0.00002
8270-SIM	Benzo(g,h,i)perylene	mg/L	--	0.000004	0.00002
8270-SIM	Chrysene	mg/L	---	0.000003	0.00002
8270-SIM	Dibenz(a,h)anthracene	mg/L	---	0.000003	0.00002
8270-SIM	Fluoranthene	mg/L	0.00296	0.000003	0.00002
8270-SIM	Fluorene	mg/L	0.05	0.000003	0.00002
8270-SIM	Indeno(1,2,3-cd)pyrene	mg/L	---	0.000002	0.00002
8270-SIM	Phenanthrene	mg/L	0.0046	0.000003	0.00002
8270-SIM	Pyrene	mg/L	0.00024	0.000003	0.00002
Sediment					
8270C-LL	Hexachlorobenzene	mg/kg	0.006[c]	0.000079	0.001
Water					
8270C-LL	Hexachlorobenzene	mg/L	0.129 [c]	0.000022	0.00022
Water					
8260B	Acrolein	mg/L	0.005	0.002	0.02
PCBs					
Soil					
8082	Aroclor-1254	mg/kg	500[d]	0.0021	0.01

Notes:

Marine/estuarine ecological benchmarks were taken from Update to Guidance for Conducting Ecological Risk Assessments at Remediation Sites in Texas RG-263 (January 2006) unless otherwise noted. When a TRRP marine value was not available, values from Buchman (2008) were used.

[a] Total chlordane.

[b] Total endrin.

[c] Buchman, M. F., 2008. NOAA Screening Quick Reference Tables, NOAA OR&R Report 08-1, Seattle WA, Office of Response and Restoration Division, National Oceanic and Atmospheric Administration.

[d] Parmelee, R. W., C.T. Phillips, R.T. Checkai, and P.J. Bohlen. 1997.Determining the Effects of Pollutants on Soil Faunal Communities and Trophic Structure Using a Refined Microcosm System. Environmental Toxicology and Chemistry, Vol. 16, No. 6, pp. 1212–1217. Value not presented in the SLERA. Value for total PCBs as congeners.

Figures

TEXAS

QUADRANGLE LOCATION

N

Scale in Feet

0 1000 2000

Source:
Base map taken from http://www.tnris.state.tx.us Freeport, Texas 7.5 min. U.S.G.S. quadrangle, 1974.

TITLE:

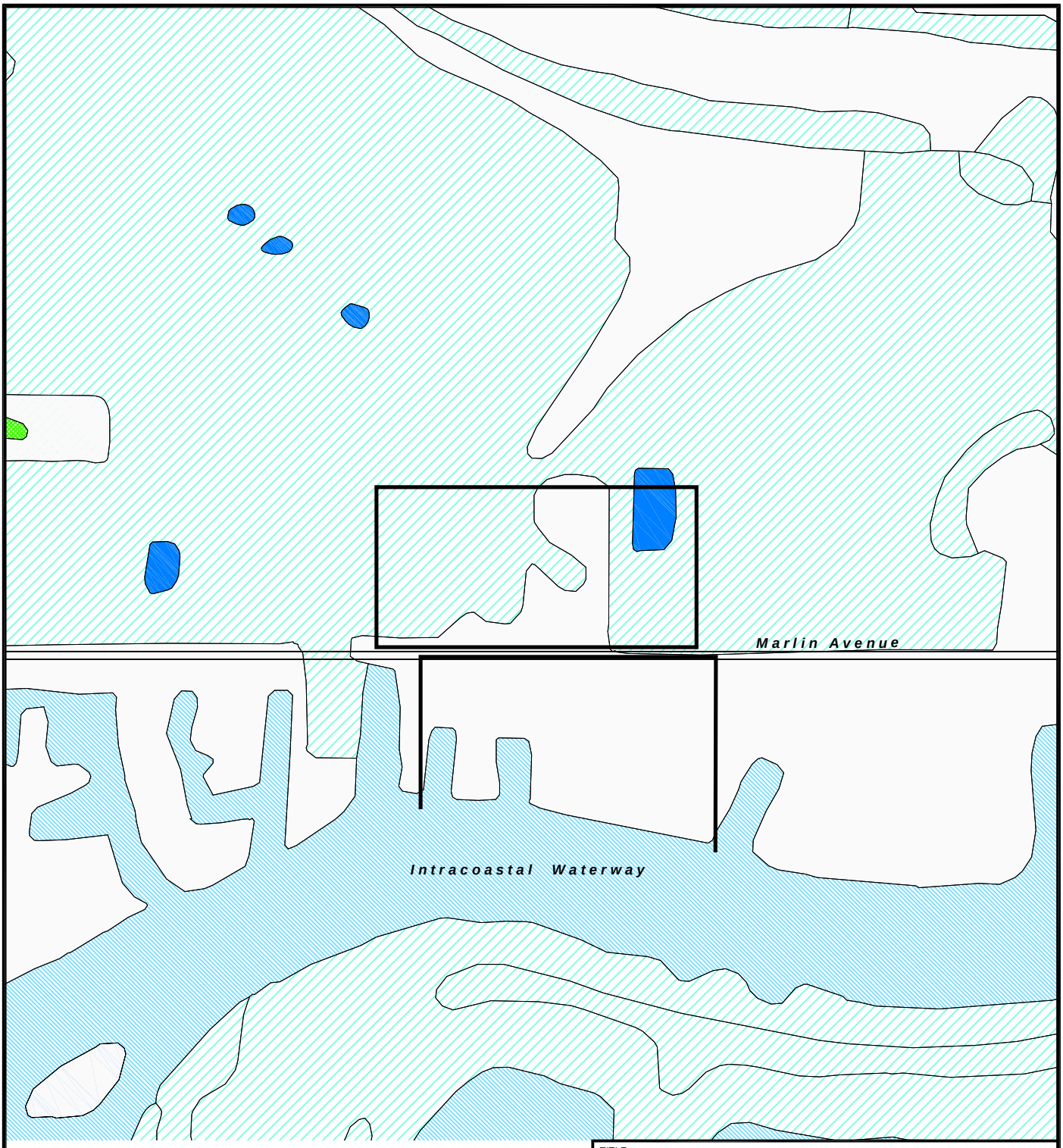
SITE LOCATION MAP

REPORT: WORK PLAN - SAMPLING AND ANALYSIS PLAN
BASELINE ECOLOGICAL RISK ASSESSMENT

SITE: GULFCO MARINE MAINTENANCE
FREEPORT, BRAZORIA COUNTY, TEXAS

PROJECT:	DRAWN:	FIGURE:
41568745	ZGK/NAB	1
DATE: MAY, 2010	CHECKED: DL	

URS
10550 RICHMOND AVE., SUITE 155
HOUSTON, TEXAS 77042
PH: 713-914-6699
FAX: 713-914-8404



EXPLANATION

- Approx. Site Boundary
- Upland Area
- Estuarine and Marine Deepwater
- Estuarine and Marine Wetland
- Freshwater Emergent Wetland
- Freshwater Pond

N

Approx. Scale in Feet

0 300 600

Source:
U.S. Fish & Wildlife Service, Wetlands Online Mapper, 2008.

TITLE:

WETLAND MAP

REPORT: **WORK PLAN - SAMPLING AND ANALYSIS PLAN BASELINE ECOLOGICAL RISK ASSESSMENT**

SITE: **GULFCO MARINE MAINTENANCE**
FREEPORT, BRAZORIA COUNTY, TEXAS

PROJECT:	41568745	DRAWN:	ZGK/NAB	FIGURE:	2
DATE:	MAY, 2010	CHECKED:	DL		

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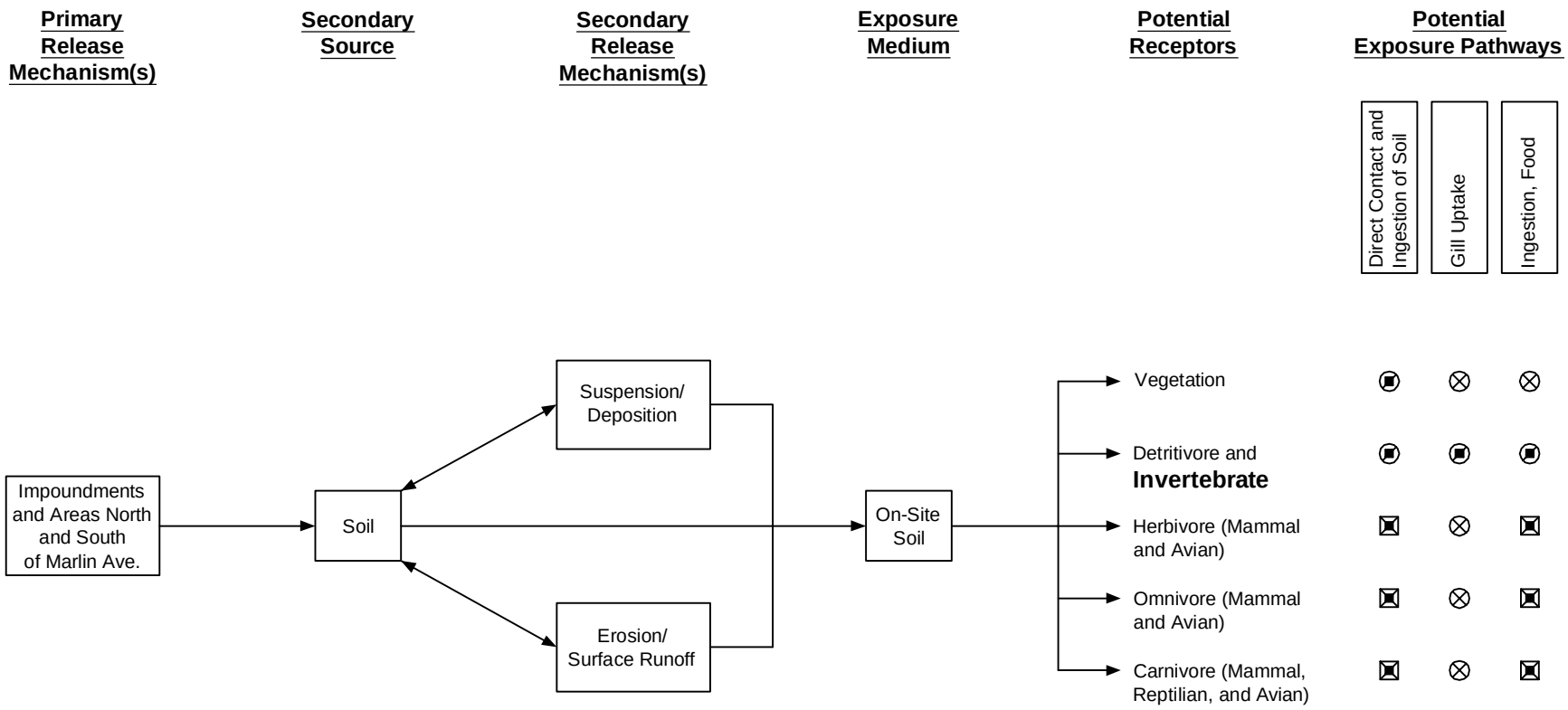
FAX: 713-914-8404



LEGEND

- ⊗ For South Area soils, pathway is mitigated by lack of complete exposure pathways. For North Area soils, pathway is potentially complete.
- ☒ No unacceptable risk (Final SLERA conclusion)
- ■ Pathway is potentially complete
- ☒ Pathway is incomplete
- ⊗ Pathway is not viable

Note:
Bolded receptors are those remaining for evaluation in the BERA after Problem Formulation refinement.

TITLE:

TERRESTRIAL ECOSYSTEM CONCEPTUAL SITE MODEL

REPORT: WORK PLAN - SAMPLING AND ANALYSIS PLAN
BASELINE ECOLOGICAL RISK ASSESSMENT

SITE: GULFCO MARINE MAINTENANCE
FREEPORT, BRAZORIA COUNTY, TEXAS

PROJECT:	DRAWN:	FIGURE:
41568745	ZGK/NAB	3
DATE: JUNE, 2010	CHECKED: DL	

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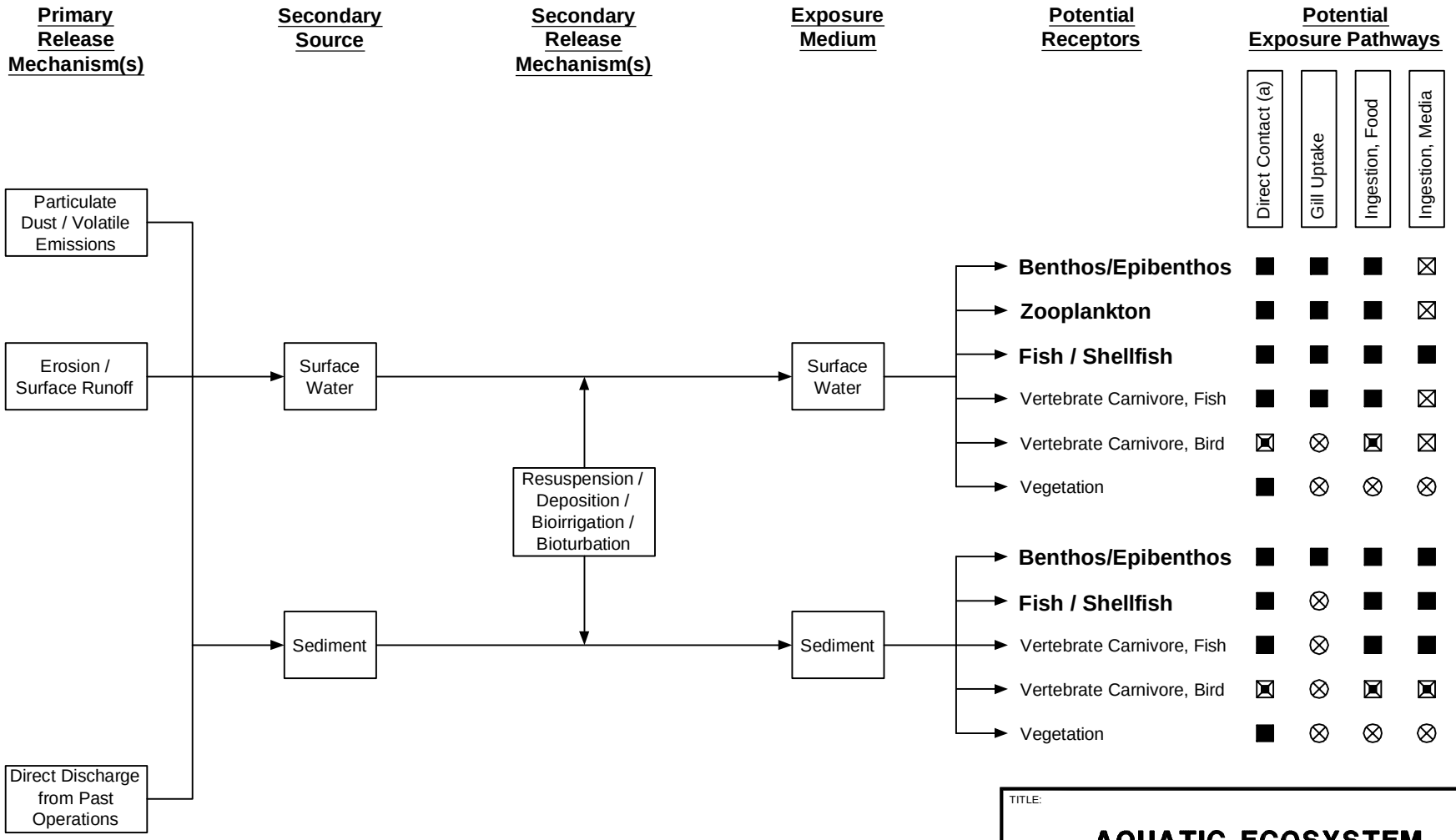

LEGEND

- ☒ No unacceptable risk (Final SLERA conclusion)
- ■ Pathway is potentially complete
- ☒ Pathway is incomplete
- ⊗ Pathway is not viable
- (a) Direct contact includes dermal absorption

Note:
Bolded receptors are those remaining for evaluation in the BERA after Problem Formulation refinement.

TITLE:

AQUATIC ECOSYSTEM CONCEPTUAL SITE MODEL

REPORT: **WORK PLAN - SAMPLING AND ANALYSIS PLAN BASELINE ECOLOGICAL RISK ASSESSMENT**

SITE: **GULFCO MARINE MAINTENANCE**
FREEPORT, BRAZORIA COUNTY, TEXAS

PROJECT:	DRAWN:	FIGURE:
41568745	ZGK/NAB	4
DATE: MAY, 2010	CHECKED: DL	

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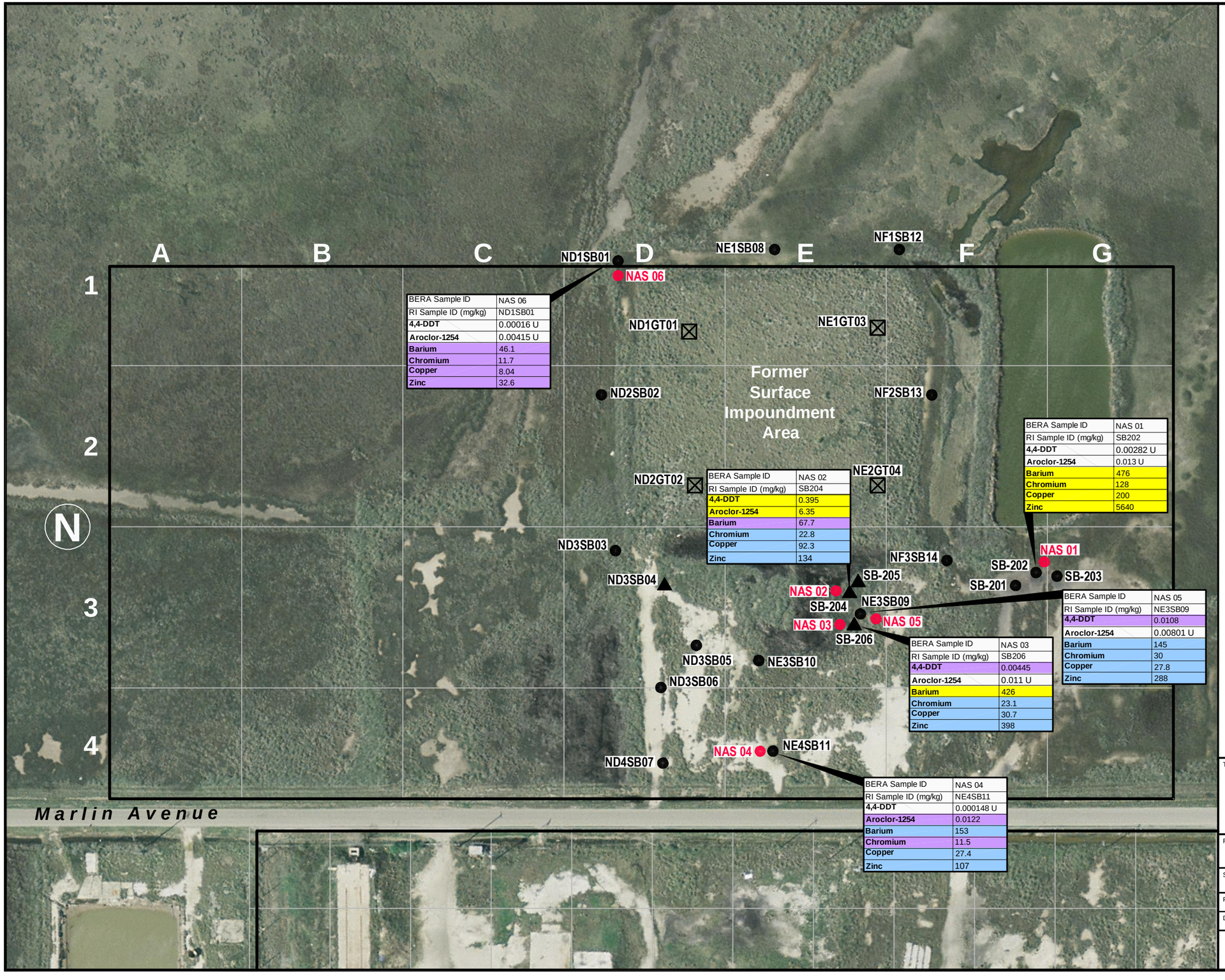

BERA Sample ID	NAS 06
RI Sample ID (mg/kg)	ND1SB01
4,4-DDT	0.00016 U
Aroclor-1254	0.00415 U
Barium	46.1
Chromium	11.7
Copper	8.04
Zinc	32.6

BERA Sample ID	NAS 01
RI Sample ID (mg/kg)	SB202
4,4-DDT	0.00282 U
Aroclor-1254	0.013 U
Barium	476
Chromium	128
Copper	200
Zinc	5640

BERA Sample ID	NAS 02
RI Sample ID (mg/kg)	SB204
4,4-DDT	0.395
Aroclor-1254	6.35
Barium	67.7
Chromium	22.8
Copper	92.3
Zinc	134

BERA Sample ID	NAS 05
RI Sample ID (mg/kg)	NE3SB09
4,4-DDT	0.0108
Aroclor-1254	0.00801 U
Barium	145
Chromium	30
Copper	27.8
Zinc	288

BERA Sample ID	NAS 03
RI Sample ID (mg/kg)	SB206
4,4-DDT	0.00445
Aroclor-1254	0.011 U
Barium	426
Chromium	23.1
Copper	30.7
Zinc	398

BERA Sample ID	NAS 04
RI Sample ID (mg/kg)	NE4SB11
4,4-DDT	0.000148 U
Aroclor-1254	0.0122
Barium	153
Chromium	11.5
Copper	27.4
Zinc	107

EXPLANATION

- Gulfco Marine Maintenance Site Boundary (approximate)
- Shallow (0-2 ft) Soil Sample
- Shallow (0-2 ft) and Deep (4+ ft) Soil Sample
- Geotechnical Soil Boring
- Proposed Sediment Sample Location

Notes:
1. BGS = below ground surface.
2. For sample concentration data, see SLERA Figures 7A through 7B.

= High Concentration
= Mid Concentration
= Low Concentration

Approx. Scale in Feet

0 60 120

Source of photo: H-GAC, Texas aerial photograph, 2006.

TITLE:

PROPOSED SAMPLE LOCATIONS NORTH AREA SOILS

REPORT: WORK PLAN - SAMPLING AND ANALYSIS PLAN BASELINE ECOLOGICAL RISK ASSESSMENT

SITE: GULFCO MARINE MAINTENANCE
FREEPORT, BRAZORIA COUNTY, TEXAS

PROJECT:	41568745	DRAWN:	ZGK/NAB	FIGURE:	5
DATE:	JUNE, 2010	CHECKED:	DL		

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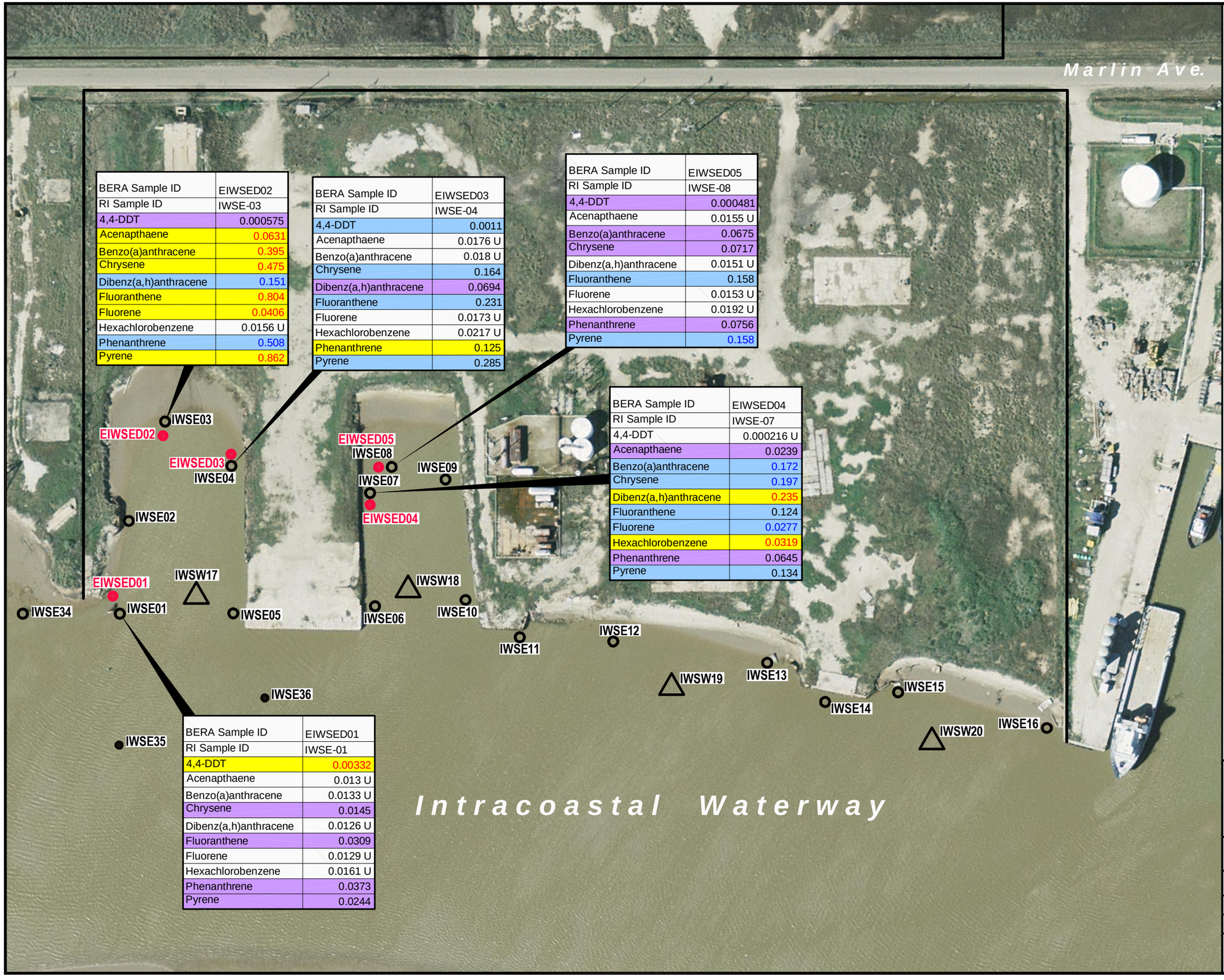

BERA Sample ID	EIWSED02
RI Sample ID	IWSE-03
4,4-DDT	0.000575
Acenapthaene	0.0631
Benzo(a)anthracene	0.395
Chrysene	0.475
Dibenz(a,h)anthracene	0.151
Fluoranthene	0.804
Fluorene	0.0406
Hexachlorobenzene	0.0156 U
Phenanthrene	0.508
Pyrene	0.862

BERA Sample ID	EIWSED03
RI Sample ID	IWSE-04
4,4-DDT	0.0011
Acenapthaene	0.0176 U
Benzo(a)anthracene	0.018 U
Chrysene	0.164
Dibenz(a,h)anthracene	0.0694
Fluoranthene	0.231
Fluorene	0.0173 U
Hexachlorobenzene	0.0217 U
Phenanthrene	0.125
Pyrene	0.285

BERA Sample ID	EIWSED05
RI Sample ID	IWSE-08
4,4-DDT	0.000481
Acenapthaene	0.0155 U
Benzo(a)anthracene	0.0675
Chrysene	0.0717
Dibenz(a,h)anthracene	0.0151 U
Fluoranthene	0.158
Fluorene	0.0153 U
Hexachlorobenzene	0.0192 U
Phenanthrene	0.0756
Pyrene	0.158

BERA Sample ID	EIWSED04
RI Sample ID	IWSE-07
4,4-DDT	0.000216 U
Acenapthaene	0.0239
Benzo(a)anthracene	0.172
Chrysene	0.197
Dibenz(a,h)anthracene	0.235
Fluoranthene	0.124
Fluorene	0.0277
Hexachlorobenzene	0.0319
Phenanthrene	0.0645
Pyrene	0.134

BERA Sample ID	EIWSED01
RI Sample ID	IWSE-01
4,4-DDT	0.00332
Acenapthaene	0.013 U
Benzo(a)anthracene	0.0133 U
Chrysene	0.0145
Dibenz(a,h)anthracene	0.0126 U
Fluoranthene	0.0309
Fluorene	0.0129 U
Hexachlorobenzene	0.0161 U
Phenanthrene	0.0373
Pyrene	0.0244

EXPLANATION

- Gulfco Marine Maintenance Site Boundary (approximate)
- RI/FS Intracoastal Waterway Sediment Sample
- RI/FS Intracoastal Waterway Surface Water Sample
- RI/FS Attempted Intracoastal Waterway Sediment Sample (not enough sediment present to allow for sampling)
- Proposed Sediment Sample Location

Notes:
1. For sample concentration data, see SLERA Figure 9.

= High Concentration
= Mid Concentration
= Low Concentration

N

Approx. Scale in Feet

0 60 120

Source of photo: H-GAC, Texas aerial photograph, 2006.

TITLE:

PROPOSED SAMPLE LOCATIONS INTRACOASTAL WATERWAY

REPORT: WORK PLAN - SAMPLING AND ANALYSIS PLAN BASELINE ECOLOGICAL RISK ASSESSMENT

SITE: GULFCO MARINE MAINTENANCE
FREEPORT, BRAZORIA COUNTY, TEXAS

PROJECT: 41568745 | DRAWN: ZGK/NAB | FIGURE: 6

DATE: JUNE, 2010 | CHECKED: DL

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FAX: 713-914-8404

EXPLANATION

- RI/FS Sediment Station
- RI/FS Surface Water Station
- Proposed Sediment Reference Sample Location

N

Approx. Scale in Feet

0 125 250

Source of photo: H-GAC, Texas aerial photograph, 2006.

TITLE:

PROPOSED SAMPLE LOCATIONS INTRACOASTAL WATERWAY REFERENCE SAMPLES

REPORT: WORK PLAN - SAMPLING AND ANALYSIS PLAN BASELINE ECOLOGICAL RISK ASSESSMENT

SITE: GULFCO MARINE MAINTENANCE
FREEPORT, BRAZORIA COUNTY, TEXAS

PROJECT:	DRAWN:	FIGURE:
41568745	ZGK/NAB	7
DATE: JUNE, 2010	CHECKED: DL	

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PH: 713-914-6699
FAX: 713-914-8404

BERA Sample ID	EWSED01
RI Sample ID	2WSED04-004
2-Methylnaphthalene	0.153 U
4,4-DDT	0.000939 U
Acenaphthene	0.153 U
Acenaphthylene	0.545
Anthracene	0.334
Arsenic	0.35 U
Benzo(a)anthracene	0.126 U
Benzo(a)pyrene	0.972
Benzo(g,h,i)perylene	1.94
Chrysene	4.05
Copper	16
Dibenz(a,h)anthracene	2.91
Endrin Aldehyde	0.00431
Endrin Ketone	0.013
Fluoranthene	0.189 U
Fluorene	0.12U
gamma-chlordane	0.0036
Indeno(1,2,3-cd)pyrene	1.94
Lead	18.3
Nickel	21.3
Phenanthrene	0.111 U
Pyrene	1.18
Zinc	116

BERA Sample ID	EWSED02
RI Sample ID	2WSED03-003
2-Methylnaphthalene	0.173 U
4,4-DDT	0.00107 U
Acenaphthene	0.173 U
Acenaphthylene	0.346
Anthracene	0.241
Arsenic	0.4 U
Benzo(a)anthracene	U
Benzo(a)pyrene	0.631
Benzo(g,h,i)perylene	1.52
Chrysene	2.73
Copper	12.6
Dibenz(a,h)anthracene	2.83
Endrin Aldehyde	0.01
Endrin Ketone	0.00619
Fluoranthene	0.213 U
Fluorene	0.135 U
gamma-chlordane	0.000862 U
Indeno(1,2,3-cd)pyrene	1.59
Lead	17.2
Nickel	20.9
Phenanthrene	0.125 U
Pyrene	0.729
Zinc	115

EXPLANATION

- Gulfco Marine Maintenance Site Boundary (approximate)
- RI/FS Sediment Sample Location
- Proposed Sediment Sample Location
- Proposed Sediment Reference Sample Location

Notes:
1. BGS = below ground surface.
2. For sample concentration data, see SLERA Figure 11.

= High Concentration
= Mid Concentration
= Low Concentration

BERA Sample ID	EWSED07
RI Sample ID	4WSED3 (0-0.5)
2-Methylnaphthalene	0.00936 U
4,4-DDT	0.00498 U
Acenaphthene	0.016
Acenaphthylene	0.00746 U
Anthracene	0.033
Arsenic	0.12 U
Benzo(a)anthracene	0.199
Benzo(a)pyrene	0.227
Benzo(g,h,i)perylene	0.209
Chrysene	0.094
Copper	27.6
Dibenz(a,h)anthracene	0.00635 U
Endrin Aldehyde	0.00579 U
Endrin Ketone	0.00527 U
Fluoranthene	0.176
Fluorene	0.015
gamma-chlordane	0.00423 U
Indeno(1,2,3-cd)pyrene	0.408
Lead	29.3
Nickel	19.6
Phenanthrene	0.135
Pyrene	0.188
Zinc	290

BERA Sample ID	EWSED03
RI Sample ID	NF4SE13-013-(0-0.5)
2-Methylnaphthalene	0.0122
4,4-DDT	0.00254
Acenaphthene	0.0103 U
Acenaphthylene	0.0117 U
Anthracene	0.0126
Arsenic	12.8
Benzo(a)anthracene	0.0106 U
Benzo(a)pyrene	0.0105 U
Benzo(g,h,i)perylene	0.133
Chrysene	0.0904
Copper	35.7
Dibenz(a,h)anthracene	0.0555
Endrin Aldehyde	0.000403 U
Endrin Ketone	0.000505 U
Fluoranthene	0.0117 U
Fluorene	0.0102 U
gamma-chlordane	0.000265 U
Indeno(1,2,3-cd)pyrene	0.0951
Lead	64.7
Nickel	27.7
Phenanthrene	0.0898
Pyrene	0.109
Zinc	903

BERA Sample ID	EWSED06
RI Sample ID	SPSE03-003
2-Methylnaphthalene	NA
4,4-DDT	0.00157
Acenaphthene	NA
Acenaphthylene	NA
Anthracene	NA
Arsenic	5.01
Benzo(a)anthracene	NA
Benzo(a)pyrene	NA
Benzo(g,h,i)perylene	0.135
Chrysene	0.0257
Copper	26.8
Dibenz(a,h)anthracene	NA
Endrin Aldehyde	NA
Endrin Ketone	NA
Fluoranthene	NA
Fluorene	NA
gamma-chlordane	NA
Indeno(1,2,3-cd)pyrene	NA
Lead	30.5
Nickel	20.6
Phenanthrene	NA
Pyrene	0.0265
Zinc	999

BERA Sample ID	EWSED05
RI Sample ID	NB4SE08-008-(0-0.5)
2-Methylnaphthalene	0.0396
4,4-DDT	0.00922
Acenaphthene	0.113
Acenaphthylene	0.0291
Anthracene	0.188
Arsenic	3.53
Benzo(a)anthracene	0.993
Benzo(a)pyrene	1.3
Benzo(g,h,i)perylene	0.862
Chrysene	1.27
Copper	39.6
Dibenz(a,h)anthracene	0.337
Endrin Aldehyde	0.00452
Endrin Ketone	0.000458 U
Fluoranthene	2.17
Fluorene	0.127
gamma-chlordane	0.00024 U
Indeno(1,2,3-cd)pyrene	1.1
Lead	88.1
Nickel	10.9
Phenanthrene	1.3
Pyrene	1.64
Zinc	601

BERA Sample ID	EWSED04
RI Sample ID	2WSED17-017 (0-0.5)
2-Methylnaphthalene	0.053
4,4-DDT	0.000829 U
Acenaphthene	0.133
Acenaphthylene	0.013 U
Anthracene	0.257
Arsenic	1.4
Benzo(a)anthracene	0.724
Benzo(a)pyrene	0.618
Benzo(g,h,i)perylene	0.527
Chrysene	0.743
Copper	25.6
Dibenz(a,h)anthracene	0.312
Endrin Aldehyde	0.000706 U
Endrin Ketone	0.000603 U
Fluoranthene	1.43
Fluorene	0.139
gamma-chlordane	0.000669 U
Indeno(1,2,3-cd)pyrene	0.752
Lead	237
Nickel	13.7
Phenanthrene	1.18
Pyrene	1.34
Zinc	404

Approx. Scale in Feet
0 75 150

Source of photo: H-GAC, Texas aerial photograph, 2006.

TITLE:

PROPOSED SAMPLE LOCATIONS WETLAND SEDIMENT

REPORT: WORK PLAN - SAMPLING AND ANALYSIS PLAN BASELINE ECOLOGICAL RISK ASSESSMENT

SITE: GULFCO MARINE MAINTENANCE
FREEPORT, BRAZORIA COUNTY, TEXAS

PROJECT: 41568745 | DRAWN: ZGK/NAB | FIGURE: 8

DATE: JUNE, 2010 | CHECKED: DL

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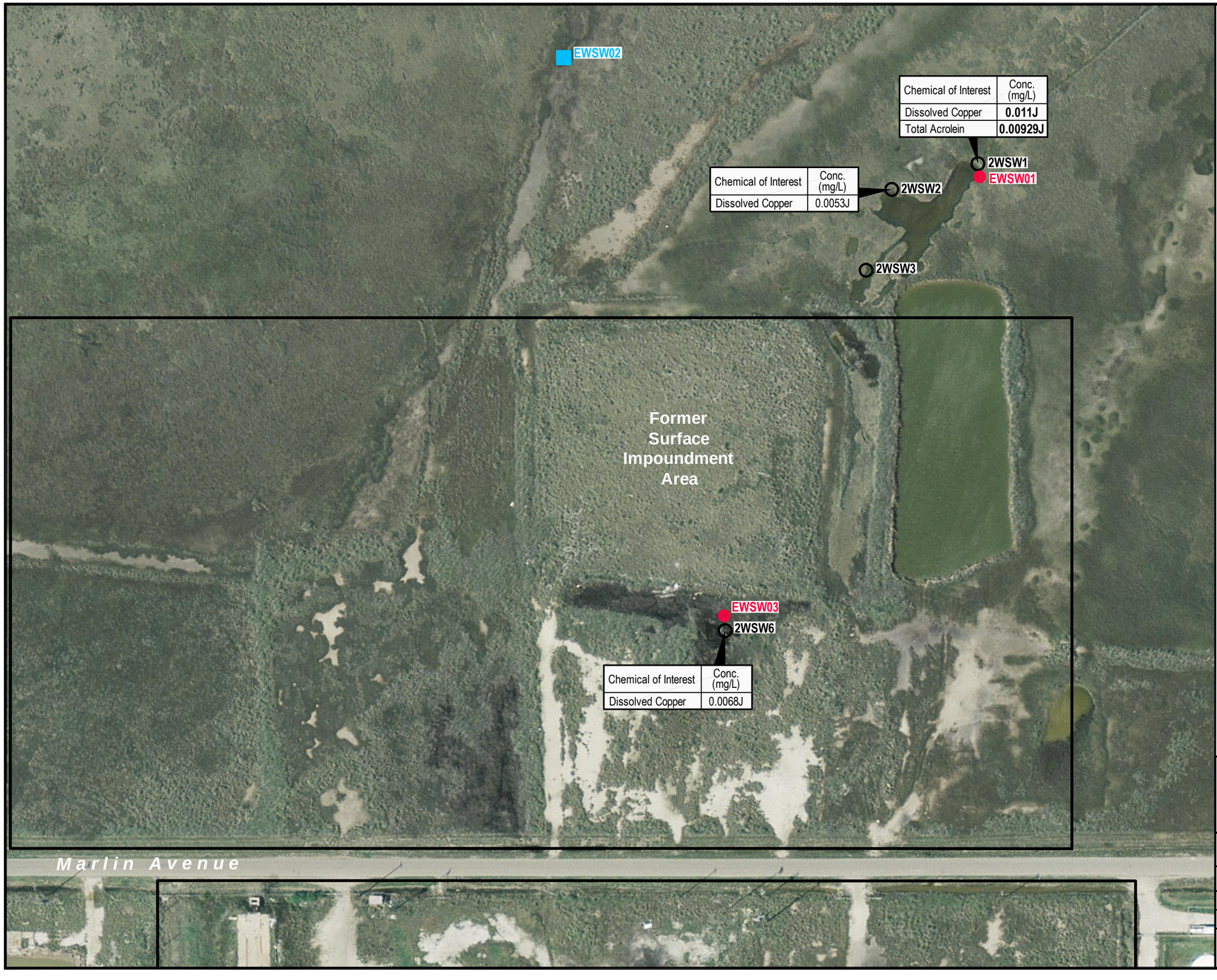

EXPLANATION

- Gulfco Marine Maintenance Site Boundary (approximate)
- RI/FS Wetland Surface Water Sample Location
- Proposed Surface Water Sample Location
- Proposed Surface Water Reference Sample Location

Notes:
1. Data Qualifier: J = Estimated value.
2. Bolded values are the maximum measured concentration for that compound.
3. Values shown in the figure exceed the Texas Surface Water Quality Standard.
4. Sample locations are subject to the presence of surface water at the time of sampling.

Approx. Scale in Feet

0 60 120

Source of photo: H-GAC, Texas aerial photograph, 2006.

TITLE:

PROPOSED SAMPLE LOCATIONS WETLAND SURFACE WATER

REPORT: WORK PLAN - SAMPLING AND ANALYSIS PLAN BASELINE ECOLOGICAL RISK ASSESSMENT

SITE: GULFCO MARINE MAINTENANCE
FREEPORT, BRAZORIA COUNTY, TEXAS

PROJECT:	DRAWN:	FIGURE:
41568745	ZGK/NAB	9
DATE: MAY, 2010	CHECKED: DL	

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Appendix A

Columbia Analytical Services Statement of Qualifications for Marine Analysis

STATEMENT OF QUALIFICATIONS





Analysis of Marine and Freshwater Sediment, Pore Water, and Tissue Samples

Columbia Analytical Services, Inc.

800.695.7222 | www.caslab.com

TABLE OF CONTENTS

I. Introduction

Since 1986, Columbia Analytical Services, Inc. (CAS) has been actively involved in the analysis of marine and freshwater sediment, water and tissue samples. Much of our analytical work is in support of dredging, remedial investigation, feasibility studies and risk assessment, which, in many cases, require extremely low-level detection limits. These types of samples present unique challenges to the laboratory due to analytical interferences caused by the matrices.

CAS has developed and implemented cleanup procedures and method modifications to specifically deal with these types of matrices. We have also developed the expertise necessary to perform complex ultra-trace analyses. These low-level analyses of sediment, tissue and water use advanced specialized instrumentation. This instrumentation includes Inductively Coupled Plasma Mass Spectroscopy (ICP/MS), purge and trap cold vapor atomic fluorescence spectrometry, High-Resolution Gas Chromatography/Mass Spectroscopy (HRGC/MS), and High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS).

CAS, headquartered in Kelso, Washington, is a certified, full-service chemical and biological analytical laboratory network. Our network is comprised of eight fixed laboratories and four service centers in Arizona, California, Florida, Hawaii, New York, Texas and Washington. In addition to supporting marine and freshwater aquatic sample analyses throughout the United States, our laboratories also possess the necessary permits to accept samples from foreign countries.

This Statement of Qualification provides a general description of CAS analytical protocols for determining trace analytes in marine and freshwater environmental samples. Detection limits for these analytes are also included. The analytes discussed in this SOQ are those typically requested for marine and freshwater projects. Also included in this SOQ, is a section discussing CAS' relevant experience that provides project references and a project experience matrix.

II. Sample Preparation

Pore Water Extraction

Pore water extractions are performed according to the latest Army Corps of Engineers (ACOE) interim protocol. CAS actively attends meetings and provides recommendations for the development of the procedures. Sample manipulations are performed in a glove box under anaerobic conditions. Double centrifuging is performed in a refrigerated centrifuge, maintaining anaerobic conditions within the sample containers. Filtration is optional, depending on project objectives. If required, filtration is performed using a silver membrane or polycarbonate filter media to prevent loss of butyltin compounds to adsorption. The analysis of pore water is performed using the procedures listed in the “Seawater” section of each constituent’s analytical protocol.

Freeze-Drying

CAS incorporates the use of freeze-drying of sediment and tissue samples for environmental analysis. Freeze-drying of sediment and tissue samples is performed prior to extraction and analysis for Polynuclear Aromatic Hydrocarbons (PAHs), Polychlorinated Biphenyls (PCBs), Pesticides, Dioxins, and Metals. The use of freeze-drying eliminates or reduces the undesirable effects of water. The most significant benefits are lower detection limits and more quantitative determinations. In addition to lower detection limits and better recoveries, freeze-drying of samples allows for complete homogenization of the sample matrices. Thus, improved precision is realized. This is particularly significant when analyzing heterogeneous samples (e.g. high organic sediments, whole-body tissues, etc.).

Tissue Homogenization

All tissue samples are subjected to homogenization techniques prior to analysis, which are designed to assure representative sub-sampling for each analytical parameter. The procedures used within CAS for homogenization vary significantly depending on the tissue type and the technical specifications for the project. Our laboratory is equipped to handle a wide variety of tissue preparations. These range from relatively simple whole body homogenization of juvenile fish, to more involved applications where small rodents require radiation treatment for destruction of biological hazards (e.g. Hantavirus, rabies, etc.) and subsequent dissection for analysis of individual organs.

Total Solids

Total solids values are derived from freeze-dried tissues. The determination is performed on a pre-homogenized wet sample. The dry solids from the freeze-drying determination are then further homogenized and used for the metals analysis (except mercury) as described in the metals section of this document. Freeze-drying is performed to avoid degradation and associated chemical changes that occur when the sample is dried at elevated temperatures.

III. Analytical Protocol

A brief description of the procedures CAS typically employs for the analysis of sediment, tissue, seawater and freshwater matrices in support of marine and freshwater studies is provided in the following sub-sections. Due to the complexity of analyzing these matrices for low-level constituents, specialized procedures beyond the scope of EPA SW 846, EPA-CLP and other routine methods are often required. Seawater presents no particular challenges when determining organic constituents. However, trace metals analysis in the presence of high dissolved solids requires relatively involved techniques to reach the levels of detection typically required to meet project objectives. CAS has been active in research and development of procedures for preparation and analyses of sediment, tissue and water samples. Our laboratory specializes in the analysis of tissue and sediment for low-level chemical constituents and has developed procedures for providing data of high technical quality that meets standard validation criteria. A summary of some of our experience over the last ten years may be found in Section IV.

A. Polyaromatic Hydrocarbons (PAHs) and Base Neutral Acid Compounds (BNAs)

Seawater and Pore Water

Sample preparations generally follow traditional solvent extraction techniques; continuous liquid/liquid or separatory funnel. These extracts rarely require cleanup procedures before instrumental analysis, and can be concentrated to smaller final volumes to gain sensitivity. For PAHs, instrumental analysis is performed using Gas Chromatograph /Mass Spectrometry (GC/MS) operated in the Selective Ion Monitoring (SIM) mode to maximize sensitivity. In addition to the standard list of PAHs typically analyzed, the associated alkylated homologs are also available. Detection limit information for the complete list of PAH compounds, including the alkylated homologs, is listed in the tables following page 13. For low-level semivolatile organic analysis conventional GC/MS techniques are used in conjunction with a Large Volume Injector (LVI) system. The LVI allows for a greater quantity of analyte to be introduced into the GC/MS. Detection limits for low-level semivolatile analytes are listed in the tables following page 13.

Sediments

Sample preparations are generally initiated using traditional solvent extraction techniques, usually soxhlet, and, occasionally, sonication. Prior to instrumental analysis, extracts are put through Gel Permeation Chromatography (GPC) cleanup and usually silica gel clean up. For PAHs, instrumental analysis is performed using Gas Chromatograph /Mass Spectrometry (GC/MS) operated in the Selective Ion Monitoring (SIM) mode to maximize sensitivity. In addition to the standard list of PAHs typically analyzed, the associated alkylated homologs are also available. Detection limit information for the complete list of PAH compounds, including the alkylated homologs, is listed in the tables following page 13. For low-level semi-volatile organic analysis conventional GC/MS techniques are used in conjunction with a Large Volume Injector (LVI) system. The LVI allows for a greater quantity of analyte to be introduced into the GC/MS than standard injection systems.

Detection limits for low-level semi-volatile analytes are listed in the tables following page 13.

Tissue

All Tissue samples are subjected to homogenization before analysis. This preparation insures representative sub-sampling for each analytical parameter. Conventional solvent extraction techniques such as soxhelt and sonication are usually employed for extracting the samples. Prior to instrumental analysis, extracts are put through Gel Permeation Chromatography (GPC) cleanup and silica gel cleanup. Removal of lipids is of particular concern during the cleanup process. The instrumental analysis is performed using GC/MS operated in SIM mode to maximize sensitivity. In addition to the standard list of PAHs typically analyzed, the associated alkylated homologs are also available. Detection limit information for the complete list of PAH compounds, including the alkylated homologs, is listed in the tables following page 13.

B. Pesticides/PCB Aroclors

Seawater and Pore Water

The pesticide and PCB Aroclor analyses are performed by following EPA Methods 8081 and 8082. Prior to instrumental analysis for pesticides, extracts are generally not put through any cleanup process. The PCB Aroclor fraction receives an acid cleanup prior to Gas Chromatograph/Electron Capture Detector (GC/ECD) analysis. Detection limit information is listed in the tables following page 13. For ultra low-level Aroclor analysis a Large Volume Injector (LVI) system is used in conjunction with GC/ECD.

Sediments

To obtain the low level detection limits required when analyzing marine sediments, the pesticide and PCB Aroclor analyses are performed by following EPA Methods 8081 and 8082 with slight modifications to the sample mass, final extract volume, and cleanup procedures. To accommodate the relatively large sample mass required to reach the low level detection limits, the samples are extracted using a sonication technique. The extracts are put through Gel Permeation Chromatography (GPC) cleanup and mercury cleanup procedures prior to splitting for Aroclor and pesticide analyses. The pesticide fraction generally goes directly to the Gas Chromatograph/Electron Capture Detector (GC/ECD) for analysis. The PCB Aroclor fraction receives an acid cleanup prior to GC/ECD analysis. Detection limit information is listed in the tables following page 13.

Tissue

To obtain the low level detection limits required when analyzing biological tissues, the pesticide and PCB Aroclor analyses are performed by following EPA Methods 8081 and 8082 with slight modifications to the sample mass, final extract volume, and cleanup procedures. In order to assure representative sub-sampling for each analytical parameter, all tissue samples are subject to homogenization prior to analysis. To accommodate the relatively large sample mass required to reach the low level detection limits, the samples are extracted using a sonication technique. The extracts are put through GPC and Florisil® cleanups prior to splitting for PCB Aroclor and pesticide analyses. The pesticide fraction generally goes directly to the GC/ECD for analysis. The PCB Aroclor fraction receives an acid cleanup prior to GC/ECD analysis. Detection limit information is listed in the tables following page 13. For ultra low-level Aroclor analysis a Large Volume Injector (LVI) system is used in conjunction with GC/ECD.

C. PCB Congeners

Seawater and Pore Water

The PCB congener analysis is performed by following EPA Method 8082 with slight modifications. The extracts are subjected to acid and permanganate cleanups prior to GC/ECD analysis. Detection limit information is listed in the tables following page 13.

Sediments

To obtain the low level detection limits required when analyzing marine sediments, the PCB congener analysis is performed by following EPA Method 8082 with slight modifications to the sample mass, final extract volume, and cleanup procedures. To accommodate the relatively large sample mass required to reach the low level detection limits, the samples are extracted using a sonication technique. The extracts are subjected to GPC, mercury, silica gel, acid, and permanganate cleanups prior to GC/ECD analysis. Detection limit information is listed in the tables following page 13.

Tissue

To obtain the low level detection limits required when analyzing biological tissues, the PCB congener analysis is performed by following EPA Method 8082 with slight modifications to the sample mass, final extract volume, and cleanup procedures. In order to assure representative sub-sampling for each analytical parameter, all tissue samples are subject to homogenization prior to analysis. To accommodate the relatively large sample mass required to reach the low level detection limits, the samples are extracted using a sonication technique. The extracts are subjected to GPC, silica gel, acid, and permanganate cleanups prior to GC/ECD analysis. Detection limit information is listed in the tables following page 13.

D. Organotin

Seawater and Pore Water

Aqueous samples are analyzed using solvent extraction, derivatization, and a Gas Chromatography Flame Photometric Detector (GC/FPD). Following the addition of surrogate compounds (tripropyltin chloride and tripentyltin chloride), aqueous samples are extracted with hexane that contains 0.2% (wt./vol.) tropolone. Extracts are derivatized with hexylmagnesium bromide in ether via a Grignard reaction. The Grignard reagent is synthesized by CAS (commercially available reagent is not used due to unacceptable purity). Extracts are cleaned by elution through alumina and silica gel columns. The extracts are analyzed by GC/FPD with a 610 nm filter. A minimum (10%) of analyte hits are confirmed by secondary column GC/FPD or GC/MS analysis. All detectable values are confirmed if the samples originated from an uncharacterized site (i.e. no historical data to suggest the likelihood of the presence of organotin). Detection limit information is listed in the tables following page 13.

Sediments

Bulk sediment samples are analyzed using solvent extraction, derivatization, and a GC/FPD. Samples are dried with muffled, anhydrous sodium sulfate. Following the addition of surrogate compounds (tripropyltin chloride and tripentyltin chloride), sediments are extracted with methylene chloride that contains 0.1% (wt./vol.) tropolone. After solvent exchange into hexane, extracts are derivatized with hexylmagnesium bromide in ether via a Grignard reaction. The Grignard reagent is synthesized by CAS (commercially available reagent is not used due to unacceptable purity). Sediment extracts are cleaned by elution through alumina and silica gel columns. The extracts are analyzed by GC/FPD with a 610 nm filter. A minimum (10%) of analyte hits are confirmed by secondary column GC/FPD or GC/MS analysis. All detectable values are confirmed if the samples originated from an uncharacterized site (i.e. no historical data to suggest the likelihood of the presence of organotin). Detection limit information is listed in the tables following page 13.

Tissue

Tissue samples are analyzed using solvent extraction, derivatization, and GC/FPD. Samples are dried with muffled, anhydrous sodium sulfate. Following the addition of surrogate compounds (tripropyltin chloride and tripentyltin chloride), tissues are extracted with methylene chloride that contains 0.1% (wt./vol.) tropolone. After solvent exchange into hexane, extracts are derivatized with hexylmagnesium bromide in ether via a Grignard reaction. The Grignard reagent is synthesized by CAS (commercially available reagent is not used due to unacceptable purity). Tissue extracts are cleaned by elution through Florisil® columns. The extracts are analyzed by GC/FPD with a 610 nm filter. A minimum (10%) of analyte hits are confirmed by a secondary column GC/FPD or GC/MS analysis. All detectable values are confirmed if the samples originated from an uncharacterized site (i.e. no historical data to suggest the likelihood of the presence of organotin). Detection limit information is listed in the tables following page 13.

E. Metals

Seawater and Pore Water

Several procedures have been used at CAS for the analysis of seawater, but the most universal technique with the best overall performance for a relatively wide range of elements is the reductive precipitation technique. The procedure incorporates a chemical separation to remove interfering matrix components so final analysis can be performed using inductively coupled plasma-mass spectroscopy (ICP-MS). The separation utilizes reduction of certain target analytes to the elemental state and precipitation of others as the boride depending on reduction potentials and/or boride solubility. The precipitation is facilitated using elemental palladium and iron boride as carriers. Once separated from the seawater matrix via centrifugation, the precipitate is re-dissolved and analyzed using ICP-MS. Typically, this procedure is performed with the intention of including arsenic and chromium in the analyses. When these elements are not of concern, some improvement of sensitivity can be achieved by altering the dissolution acid used in the procedure. Detection limit information is listed in the tables following page 13. Mercury determinations are generally performed using EPA Method 1631, purge and trap atomic fluorescence. Detection limit information is listed in the tables following page 13.

Sediments

Sediment samples are prepared for analysis using one of two approaches. One procedure incorporates the use of hydrofluoric acid to assure dissolution of refractory compounds and/or refractory compounds containing heavy metals (i.e. contained within the crystalline structure). In recent years, this approach has almost been eliminated for marine studies conducted for environmental applications. Currently, the digestion procedure most commonly required consists of a more traditional nitric/peroxide dissolution essentially equivalent to the EPA soil procedures. CAS performs both procedures. The analysis of the digestate for trace constituents is typically performed using ICP-MS. Major components are analyzed using ICP-Optical Emission Spectrometry (OES). Sediment samples generally present no analytical difficulties with regard to uncorrectable interferences. Occasionally, Graphite Furnace Atomic Absorption Spectrophotometry (GFAAS) is needed for confirmation of some elements. Detection limit information is listed in the tables following page 13.

For mercury, a larger aliquot of the wet sample is digested than is usually done for routine analyses of solid and semi-solid materials. This allows representative sub-sampling of sediments and provides the additional sensitivity typically required. The digestion procedure incorporates similar ratios of digesting/oxidizing reagents as standard EPA procedures. Additional concentrated nitric is added to facilitate the digestion of the high organic content. Standard Cold Vapor Atomic Absorption Spectrophotometry (CVAAS) technique is used for the analysis of the digestate. Detection limit information is listed in the tables following page 13.

Tissue

The digestion procedure for all elements except mercury consists of an acid digestion-oxidation under elevated temperature and pressure in a closed system. The procedure is generally preferred over modifications to conventional EPA soil digestions for several reasons. By freeze-drying the sample and grinding it to a homogenous meal, a representative sample is easily obtained. This is especially significant when analyzing whole-body samples where bone, gristle, and skin are difficult to disperse uniformly throughout the sample. This is also true for portions of bivalve samples that are very difficult to homogenize when wet. Besides helping homogeneity, the absence of water in freeze-drying facilitates the digestion/oxidation of organic material by the oxidants added. Performing the digestion in a closed Teflon vessel under elevated temperature and pressure also increases the completeness of digestion and minimizes loss of target analytes during the procedure (i.e. superior matrix spike recoveries are attained).

For mercury, our laboratory digests a larger aliquot of the wet sample than is typically done for routine analyses of solid and semi-solid materials. This allows representative sub-sampling of tissues. The digestion procedure incorporates similar ratios of digesting/oxidizing reagents as standard EPA procedures. Additional concentrated nitric is added to facilitate the digestion of the high organic content.

The digestates are analyzed using a combination of ICP-MS, ICP-OES, GFAAS, and CVAAS. Selenium is typically analyzed using GFAAS because of uncorrectable isobaric interferences when using ICP-MS. Mercury is analyzed in tissue using standard cold vapor techniques. Our laboratory does perform ultra trace mercury determinations using purge and trap cold vapor atomic fluorescence techniques, but generally does not need the added sensitivity to obtain the required detection limits to meet most project objectives. All other elements are analyzed using ICP-MS or ICP-OES, depending on the required sensitivity. Detection limit information is listed in the tables following page 13.

F. Dioxins/Furans

Seawater and Pore Water

The polychlorinated dioxins/furans analyses are performed by EPA Methods 8290 and 1613 to meet part per quadrillion detection limits usually specified for this work. The typical reporting limits are listed in the tables following page 13. In order to reach these ultra-low detection limits, extensive procedures were developed to minimize contamination. These procedures minimize sample transfer and use disposable glassware where feasible.

Sediments

CAS follows EPA Methods 8280, 8290, and 1613 to perform dioxin/furan analyses. EPA Methods 8290 and 1613 require high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) techniques to meet the parts per trillion (sediment) detection limits typically requested. The reporting limits are listed in the tables following page 13. In order to reach these ultra-low detection limits, extensive procedures were developed to minimize contamination. These procedures minimize sample transfer and use disposable glassware where feasible. Special clean-up techniques have been specifically developed for sediment to minimize matrix interferences.

Tissue

Analysis is performed by EPA Methods 8280, 8290, and 1613 on biological tissue samples. Special clean-up techniques were developed for dealing with tissue samples verses sediment samples to remove biologically active components that could interfere with the analysis. Instrumental analysis is performed by HRGC/HRMS techniques to meet the one part-per-trillion detection limit often requested for tissue samples. Typical reporting limits are listed in the tables following page 13.

Dioxin/Furan Screening

CAS provides full service dioxin testing. In our Houston laboratory both high and low resolution GCMS methodologies are performed on a variety of sample matrices: XAD resins/filters, sediments, tissues, paper, ash, soil, water, and waste. Methodologies employed by CAS/Houston include: EPA 8290, EPA 8280, EPA 613, EPA 1613, and NCASI 551.

IV. Experience

Since 1986, Columbia Analytical Services, Inc. (CAS) has been actively involved in the analysis of water, sediment and tissue in support investigations of sediments and dredge spoils as administered by the Army Corps of Engineers, the US EPA, Port Authorities and various other government agencies throughout the US and other countries. CAS has performed chemical analyses in support of the Puget Sound Estuary Program (PSEP), Puget Sound Dredged Disposal Analyses (PSDDA), and the Puget Sound Water Quality Authority. These studies have included numerous analyses of sediment, tissue and water samples for a variety of trace metals, organics, and conventional chemical constituents. Specific project experience is discussed in the following paragraphs and listed in the following experience matrix.

Sediment Testing: Our project work involves the development and validation of specialized analytical techniques to meet the low-level detection limits and difficult matrix requirements of sediment samples. All data generated under sediment programs must meet specific quality control and stringent data deliverable requirements for complete data validation.

Tissue: CAS performs trace level analyses of a variety of marine tissues. Typical matrices are marine and freshwater fish, as well as crustaceans, mollusks and other invertebrates. Project work involves developing and validating specialized analytical techniques to meet difficult matrix and low-level detection limit requirements. This includes the development of dissection and other sample preparation techniques as well as sample digestion procedures.

Ultra-Trace Metals: CAS performs ultra-trace level metals analyses of pore water samples associated with harbor dredging projects. The analyses can be extremely challenging due to the sample matrix and the limited volume of sample available. Detection limits in the sub-part per billion (ppb) range are commonly requested and the analyses are supported by strict QA/QC protocols.

CAS EXPERIENCE MATRIX

Most of these projects have typically required validatable data packages, including project-specific data deliverables.

	Regulatory Programs								Technical Elements																
	CERCLA	Washington SMS	Washington PSDDA/PSEP	EPA Green/Gold Book	Clean Water Act (TMDL, 404)	Regional Board Protocols	NOAA Status and Trends	Regional Regulatory Program	Methods Development	Physical Sediment Properties	Metals Analysis	Semivolatiles Analysis (A/B/N)	PCB Aroclors	PCB Congeners	Ultra-Low Level Analysis	Volatiles Analysis	Organotins Analysis	Organochlorine Pesticides	Lipids	Petroleum Hydrocarbons	AVS	SEM	Screen PAHs, PCBs, Dioxins, or Dioxin/Furans	TOC
Alaska Mine Discharge and Investigations *(analysis of sediment, soil, freshwater, & other samples)*				•		•		•		•	•	•	•		•	•		•		•				•
Alaska Pulp Corp. RI/FS *(analysis of sediment, marine, & wood samples)*	•									•	•	•	•			•		•		•	•	•		•
Alaska River Bioaccumulation Study *(analysis of tissue, sediment & freshwater samples)*					•			•	•	•	•	•	•	•	•			•	•					•
Columbia River Estuary Study Task Force Studies *(analysis of tissue, sediment, soil, freshwater, porewater)*					•			•		•	•	•	•	•		•	•	•	•	•				•
Coos Bay Investigations *(analysis of tissue, sediment, marine water, porewater, & wood-related materials)*	•							•	•	•	•	•	•			•	•	•		•				•
Duwamish River Sediment Characterization *(analysis of sediment samples)*		•						•	•	•	•				•		•		•					
Duwamish River Water Quality Assessment *(analysis of marine water & freshwater samples)*								•					•	•	•									
East Waterway Bioaccumulation Testing *(analysis of tissue, sediment & freshwater samples)*				•				•							•		•		•					
Forest Service Abandoned Mine Investigations *(analysis of sediment, soil, freshwater samples)*	•							•		•	•	•	•			•		•		•				•
Freshwater Stream Biota Toxics Inventory *(analysis of tissue, sediment, soil, & freshwater sample)*							•	•		•	•	•	•		•	•		•	•	•				•
Grand Calumet PRP Analytical Support *(analysis of sediment & freshwater samples)*	•							•	•	•			•		•			•		•				•
Hugo Neu-Proler Sediment Investigation *(analysis of sediment & marine water samples)*						•				•	•	•	•			•		•		•				•
Hylebos Waterway Cleanup Committee Investigations *(analysis of sediment & wood-related materials)*		•						•			•											•		•
Hylebos Waterway Wood Debris Group Cleanup *(analysis of sediment & wood-related materials)*		•									•	•												•
Hylebos Waterway Wood Debris Group Cleanup *(analysis of sediment & marine water samples)*	•							•				•												•
Jackson Park Housing Complex RI/FS *(analysis of sediment & soil samples)*	•									•	•	•	•			•		•			•	•		•
Ketchikan Pulp Superfund Investigation *(analysis of sediment, marine water & wood-related materials)*	•			•						•	•	•	•			•		•		•				•
Marina Sediment Characterization *(analysis of sediment & freshwater samples)*		•	•					•		•	•	•	•			•	•	•		•	•	•		•
McCormick and Baxter Creosoting CompanyRI/FS *(analysis of sediment, soil, freshwater and wood-related samples)*								•		•	•	•	•			•		•		•				•
Midway California Sediment Investigation *(analysis of sediment, marine water & freshwater samples)*						•		•		•	•	•	•			•		•		•	•			•
NCASI Freshwater and Marine Studies *(analysis of tissue, sediment, soil, marine water, freshwater and wood-related samples)*								•		•	•	•	•		•	•		•						•
NOAA-NMFS Overflow Analytical Support *(analysis of tissue samples)*							•				•	•	•	•	•			•	•					
Port Arthur Sediment RI *(analysis of sediment, marine water & freshwater samples)*	•									•					•		•				•	•		•
Port of Kalama Investigations *(analysis of sediment, freshwater & porewater samples)*		•						•		•	•	•	•			•		•		•				•
Port of LA Operable Unit 2&3 *(analysis of sediment & marine water samples)*				•		•				•	•	•	•			•	•	•		•				•
Port of Newport Dredge Characterization *(analysis of sediment & marine water samples)*								•		•	•	•	•			•	•	•		•				•
Port of Portland General Environmental Services *(analysis of tissue, sediment, soil, porewater, freshwater and other samples)*	•			•				•		•	•	•	•			•	•	•		•				•
Port of San Diego- Analytical Services *(analysis of sediment, marine water & freshwater samples)*						•				•	•	•	•			•	•	•		•				•

CAS EXPERIENCE MATRIX

Most of these projects have typically required validatable data packages, including project-specific data deliverables.

	Regulatory Programs								Technical Elements																
	CERCLA	Washington SMS	Washington PSDDA/PSEP	EPA Green/Gold Book	Clean Water Act (TMDL, 404)	Regional Board Protocols	NOAA Status and Trends	Regional Regulatory Program	Methods Development	Physical Sediment Properties	Metals Analysis	Semivolatiles Analysis (A/B/N)	PCB Aroclors	PCB Congeners	Ultra-Low Level Analysis	Volatiles Analysis	Organotins Analysis	Organochlorine Pesticides	Lipids	Petroleum Hydrocarbons	AVS	SEM	Screen PAHs, PCBs, Dioxins, or Dioxin/Furans	TOC
Port of San Diego- Chula Vista Dredge *(analysis of sediment samples)*						•				•	•	•	•				•	•						•
Port of Seattle T-3 *(analysis of tissue, sediment, soil, marine water, porewater)*		•	•					•		•	•	•	•	•		•	•	•	•	•	•	•		•
Portland Shipyard RI/FS *(analysis of sediment, soil, marine water & freshwater samples)*				•				•	•	•	•	•	•			•	•	•		•	•			•
Potlatch Sediment and Effluent Studies *(analysis of sediment, soil, freshwater, wood-related and other samples)*					•			•		•	•	•	•			•		•		•				•
Puget Sound Confined Disposal Site Study *(analysis of tissue, sediment & marine water samples)*		•	•					•		•	•	•	•			•	•	•	•	•	•	•		•
Rayonier Mill and Landfill Analytical Services *(analysis of sediment, soil, freshwater, wood-related and other samples)*		•						•		•	•	•	•			•		•		•	•			•
Ross Island Initial and RI *(analysis of tissue, sediment, soil, porewater, freshwater and wood-related samples)*								•	•	•	•	•	•			•	•	•	•	•	•	•		•
San Francisco Corps Sediment Monitoring *(analysis of sediment, marine water & freshwater samples)*				•				•		•	•	•	•			•	•	•						•
South Carolina Superfund Investigation *(analysis of tissue, sediment, marine water & freshwater samples)*	•									•	•	•	•	•		•	•	•	•		•			•
Spokane River Investigation *(analysis of tissue, sediment, soil, porewater, freshwater and wood-related samples)*					•			•	•	•	•	•	•	•	•	•		•	•	•				•
Tongue Point Finger Piers and Landfill RI *(analysis of sediment & marine water samples)*	•									•	•	•	•					•		•				•
Totem Marine Sediment Investigation *(analysis of sediment samples)*		•								•	•	•	•			•	•	•						•
Tributyl Tin Method Porewater Development Study *(analysis of marine water & freshwater samples)*	•		•					•	•	•		•			•		•							
U.S. EPA SAS Program- Tissue Studies *(analysis of tissue, sediment, soil, marine water, freshwater, porewater, air & other samples)*	•			•					•	•	•	•	•	•		•		•	•	•	•	•		•
U.S. Oil & Refining PSDDA Characterization *(analysis of sediment samples)*		•	•						•	•	•	•	•			•	•	•		•	•			•
U.S. Navy Pearl Harbor/West Loch Dredge *(analysis of sediment, porewater, and tissue samples)*											•	•		•			•	•			•		•	•
U.S. Navy Puget Sound Long Term Monitoring *(analysis of tissue, sediment & marine water samples)*		•	•	•						•	•	•	•			•	•	•	•	•	•	•		•
U.S. Navy San Diego Bay Sediment and Toxicity Analysis *(analysis of tissue, sediment & marine water samples)*						•		•		•	•	•		•	•	•		•	•					•
NOAA BioEffects and Status and Trends Programs Sediment samples from the areas below were tested by P450HRGS (EPA4425) Southern Calif. Bays Galveston Bay, Biscayne Bay and Sabine Lake, Texas St. Lucie Bay, Florida Northern, Central and Southern Puget Sound Charleston Harbor and Winyah Bay, South Carolina Delaware River and Bay Chesapeake Bay 1998, 1999 and 2001 San Francisco Bay 2000 and 2001 San Diego Bay																							•	
U. S. ACE - Columbia and Willamette Rivers Sediment samples from the areas below were tested by P450HRGS (EPA4425)																							•	
U. S. ACE - Miami Harbor Expansion & Maintenance Dredging (Analysis of sediment and tissue samples)											•				•			•	•	•				
Southern CA Coastal Water Res. Project - So. CA Bight 1998 Sediment samples from the area below were tested by P450HRGS (EPA4425)																							•	

TABLE 1

Polynuclear Aromatic Hydrocarbons (PAHs) by
Gas Chromatography/Mass Spectrometry (GC/MS) Selected Ion Monitoring (SIM)
Method Detection Limits (MDLs) and Method Reporting Limits (MRLs)

	Water (ug/L)		Soil/Sediment (µg/Kg) (Dry Wt. Basis)		Tissue (µg/Kg) (Wet Wt. Basis)	
Analyte	**MDL**	**MRL**	**MDL**	**MRL**	**MDL**	**MRL**
Naphthalene	0.004	0.02	0.3	5	0.3	5
2-Methylnaphthalene	0.004	0.02	0.3	5	0.2	5
1-Methylnaphthalene	0.004	0.02	0.2	5	0.2	5
C2-Naphthalenes*	0.02	0.02	5	5	5	5
C3-Naphthalenes*	0.02	0.02	5	5	5	5
C4-Naphthalenes*	0.02	0.02	5	5	5	5
Acenaphthylene	0.002	0.02	0.2	5	0.05	5
Acenaphthene	0.003	0.02	0.3	5	0.08	5
Dibenzofuran	0.003	0.02	0.2	5	0.06	5
Fluorene	0.003	0.02	0.2	5	0.06	5
C1-Fluorenes*	0.02	0.02	5	5	5	5
C2-Fluorenes*	0.02	0.02	5	5	5	5
C3-Fluorenes*	0.02	0.02	5	5	5	5
Dibenzothiophene	0.003	0.02	0.2	5	0.2	5
C1-Dibenzothiophenes*	0.02	0.02	5	5	5	5
C2-Dibenzothiophenes*	0.02	0.02	5	5	5	5
C3-Dibenzothiophenes*	0.02	0.02	5	5	5	5
Phenanthrene	0.003	0.02	0.2	5	0.07	5
Anthracene	0.003	0.02	0.2	5	0.06	5
C1-Phenanthrenes/Anthracenes*	0.02	0.02	5	5	5	5
C2-Phenanthrenes/Anthracenes*	0.02	0.02	5	5	5	5
C3-Phenanthrenes/Anthracenes*	0.02	0.02	5	5	5	5
C4-Phenanthrenes/Anthracenes*	0.02	0.02	5	5	5	5
Fluoranthene	0.003	0.02	0.2	5	0.06	5
Pyrene	0.003	0.02	0.2	5	0.07	5
C1-Fluoranthenes/Pyrenes*	0.02	0.02	5	5	5	5
Benz(a)anthracene	0.003	0.02	0.2	5	0.06	5
Chrysene	0.003	0.02	0.2	5	0.08	5
C1-Chrysenes*	0.02	0.02	5	5	5	5
C2-Chrysenes*	0.02	0.02	5	5	5	5
C3-Chrysenes*	0.02	0.02	5	5	5	5
C4-Chrysenes*	0.02	0.02	5	5	5	5
Benzo(b)fluoranthene	0.002	0.02	0.2	5	0.05	5
Benzo(k)fluoranthene	0.004	0.02	0.2	5	0.09	5
Benzo(a)pyrene	0.002	0.02	0.2	5	0.08	5
Indeno(1,2,3-cd)pyrene	0.002	0.02	0.2	5	0.08	5
Dibenz(a,h)anthracene	0.003	0.02	0.2	5	0.08	5
Benzo(g,h,i)perylene	0.004	0.02	0.1	5	0.1	5

* Method Detection Limits have not been experimentally determined for these analytes. The MDL listed is used for reporting purposes and is equal to the MRL.

Note: Lower detection limits in water are available. Please call laboratory for specifics.

6/16/04

TABLE 2

Polynuclear Aromatic Hydrocarbons (PAHs) by

Gas Chromatography/Mass Spectrometry (GC/MS) Selected Ion Monitoring (SIM)

ULTRA LOW LEVEL

Method Detection Limits (MDLs) and Method Reporting Limits (MRLs)

	Sediment (µg/Kg) (Dry Wt. Basis)		Tissue (µg/Kg) (Wet Wt. Basis)	
Analyte	**MDL**	**MRL**	**MDL**	**MRL**
Naphthalene	0.2	1	0.3	1
2-Methylnaphthalene	0.2	1	0.2	1
1-Methylnaphthalene	0.2	1	0.2	1
Acenaphthylene	0.2	0.5	0.05	0.5
Acenaphthene	0.3	0.5	0.08	0.5
Dibenzofuran	0.2	0.5	0.06	0.5
Fluorene	0.2	0.5	0.06	0.5
Dibenzothiophene	0.2	0.5	0.2	0.5
Phenanthrene	0.2	0.5	0.07	0.5
Anthracene	0.2	0.5	0.06	0.5
Fluoranthene	0.2	0.5	0.06	0.5
Pyrene	0.2	0.5	0.07	0.5
Benz(a)anthracene	0.2	0.5	0.06	0.5
Chrysene	0.2	0.5	0.08	0.5
Benzo(b)fluoranthene	0.2	0.5	0.05	0.5
Benzo(k)fluoranthene	0.2	0.5	0.09	0.5
Benzo(a)pyrene	0.2	0.5	0.08	0.5
Indeno(1,2,3-cd)pyrene	0.2	0.5	0.08	0.5
Dibenz(a,h)anthracene	0.2	0.5	0.08	0.5
Benzo(g,h,i)perylene	0.1	0.5	0.1	0.5

Note: Lower detection limits in water are available. Please call laboratory for specifics.

TABLE 3

Low Level Semivolatile Organic Compounds

Gas Chromatography/Mass Spectrometry using Large Volume Injector (LVI)

Method Detection Limits (MDLs) and Method Reporting Limits (MRLs)

	Water (µg/L)		Soil/Sediment (µg/Kg) (Dry Wt. Basis)	
Analyte	**MDL**	**MRL**	**MDL**	**MRL**
1,2,4-Trichlorobenzene	0.02	0.2	2	10
1,2-Dichlorobenzene	0.02	0.2	2	10
1,3-Dichlorobenzene	0.02	0.2	2	10
1,4-Dichlorobenzene	0.02	0.2	2	10
2,4,6-Trichlorophenol	0.04	0.5	2	10
2,4-Dichlorophenol	0.03	0.5	2	10
2,4-Dimethylphenol	0.4	2	6	50
2,4-Dinitrophenol	0.6	4	40	200
2,4-Dinitrotoluene	0.02	0.2	3	10
2,6-Dinitrotoluene	0.009	0.2	3	10
2-Chloronaphthalene	0.02	0.2	4	10
2-Chlorophenol	0.02	0.5	2	10
2-Methyl-4,6-dinitrophenol	0.02	2	2	100
2-Methylnaphthalene	0.02	0.2	2	10
2-Methylphenol	0.06	0.5	4	10
2-Nitroaniline	0.02	0.2	3	20
2-Nitrophenol	0.02	0.5	3	10
3- and 4-Methylphenol Coelution	0.06	0.5	3	10
3,3'-Dichlorobenzidine	0.5	2	4	100
3-Nitroaniline	0.3	1	3	20
4-Bromophenyl Phenyl Ether	0.020	0.2	2	10
4-Chloro-3-methylphenol	0.03	0.5	3	10
4-Chloroaniline	0.02	0.2	3	10
4-Chlorophenyl Phenyl Ether	0.009	0.2	2	10
4-Methylphenol	0.06	0.5	3	10
4-Nitroaniline	0.2	1	4	20
4-Nitrophenol	0.6	2	30	100
Acenaphthene	0.009	0.2	1	10
Acenaphthylene	0.02	0.2	2	10
Anthracene	0.02	0.2	2	10
Azobenzene	0.02	0.2	3	10
Benz(a)anthracene	0.02	0.2	2	10
Benzo(a)pyrene	0.02	0.2	2	10
Benzo(b)fluoranthene	0.02	0.2	3	10
Benzo(g,h,i)perylene	0.02	0.2	3	10
Benzo(k)fluoranthene	0.02	0.2	3	10
Benzoic Acid	2	5	96	200
Benzyl Alcohol	1	5	4	10

TABLE 3 - CONTINUED

Low Level Semivolatile Organic Compounds

Gas Chromatography/Mass Spectrometry using Large Volume Injector (LVI)

Method Detection Limits (MDLs) and Method Reporting Limits (MRLs)

	Water (µg/L)		Soil/Sediment (µg/Kg) (Dry Wt. Basis)	
Analyte	**MDL**	**MRL**	**MDL**	**MRL**
Bis(2-chloroethoxy)methane	0.02	0.2	2	10
Bis(2-chloroethyl) Ether	0.02	0.2	3	10
Bis(2-chloroisopropyl) Ether	0.02	0.2	2	10
Bis(2-ethylhexyl) Phthalate	0.3	2	2	200
Butyl Benzyl Phthalate	0.03	0.2	2	10
Carbazole	0.02	0.2	2	10
Chrysene	0.02	0.2	2	10
Dibenz(a,h)anthracene	0.04	0.2	3	10
Dibenzofuran	0.02	0.2	2	10
Diethyl Phthalate	0.03	0.2	4	10
Dimethyl Phthalate	0.02	0.2	2	10
Di-n-butyl Phthalate	0.03	0.2	3	10
Di-n-octyl Phthalate	0.04	0.2	2	10
Fluoranthene	0.02	0.2	3	10
Fluorene	0.02	0.2	2	10
Hexachlorobenzene	0.02	0.2	3	10
Hexachlorobutadiene	0.02	0.2	2	10
Hexachlorocyclopentadiene	0.05	1	15	50
Hexachloroethane	0.02	0.2	3	10
Indeno(1,2,3-cd)pyrene	0.03	0.2	2	10
Isophorone	0.009	0.2	2	10
Naphthalene	0.02	0.2	2	10
Nitrobenzene	0.008	0.2	2	10
N-Nitrosodi-n-propylamine	0.04	0.2	4	10
N-Nitrosodiphenylamine	0.03	0.2	3	10
Pentachlorophenol (PCP)	0.03	1	9	50
Phenanthrene	0.02	0.2	2	10
Phenol	0.02	0.5	2	30
Pyrene	0.02	0.2	2	10

TABLE 4

Organochlorine Pesticides
Gas Chromatography (GC), EPA Method 8081
Method Detection Limits (MDLs) & Method Reporting Limits (MRLs)

	Water (µg/L)		Soil/Sediment (µg/Kg) (Dry Wt. Basis)		Tissue (µg/Kg) (Wet Wt. Basis)	
Analyte	**MDL**	**MRL**	**MDL**	**MRL**	**MDL**	**MRL**
alpha-BHC	0.001	0.01	0.1	1	0.2	1
beta-BHC	0.003	0.01	0.2	1	0.2	1
gamma-BHC (Lindane)	0.001	0.01	0.1	1	0.3	1
delta-BHC	0.002	0.01	0.1	1	0.3	1
Heptachlor	0.001	0.01	0.1	1	0.5	1
Aldrin	0.001	0.01	0.3	1	0.2	1
Heptachlor Epoxide	0.001	0.01	0.1	1	0.2	1
gamma-Chlordane	0.001	0.01	0.04	1	0.1	1
Endosulfan I	0.001	0.01	0.1	1	0.1	1
alpha-Chlordane	0.003	0.01	0.1	1	0.4	1
Dieldrin	0.001	0.01	0.1	1	0.1	1
4,4'-DDE	0.001	0.01	0.1	1	0.1	1
Endrin	0.001	0.01	0.2	1	0.1	1
Endosulfan II	0.001	0.01	0.1	1	0.4	1
4-4'-DDD	0.002	0.01	0.09	1	0.1	1
Endrin Aldehyde	0.002	0.01	0.2	1	0.2	1
Endosulfan Sulfate	0.003	0.01	0.2	1	0.3	1
4,4'-DDT	0.001	0.01	0.2	1	0.4	1
Endrin Ketone	0.001	0.01	0.06	1	0.3	1
Methoxychlor	0.001	0.01	0.2	1	0.3	1
Toxaphene	0.04	0.5	7	50	6	50
NOAA List						
Hexachlorobenzene	0.0006	0.01	0.2	1	0.3	1
Chlorpyrifos	0.002	0.01	0.06	1	0.2	1
Oxychlordane	0.0009	0.01	0.1	1	0.1	1
2,4'-DDE	0.0009	0.01	0.07	1	0.1	1
trans-Nonachlor	0.002	0.01	0.03	1	0.05	1
2,4'-DDD	0.0008	0.01	0.16	1	0.2	1
cis-Nonachlor	0.003	0.01	0.04	1	0.1	1
2,4'-DDT	0.001	0.01	0.08	1	0.2	1
Mirex	0.0009	0.01	0.06	1	0.3	1

TABLE 4 - CONTINUED

Organochlorine Pesticides (Ultra Lowl Level)
Gas Chromatography (GC), EPA Method 8081
Method Detection Limits (MDLs) & Method Reporting Limits (MRLs)

	Water (ng/L)	
Analyte	**MDL**	**MRL**
alpha-BHC	0.3	0.5
beta-BHC	*	0.5
gamma-BHC (Lindane)	0.2	0.5
delta-BHC	0.06	0.5
Heptachlor	0.07	0.5
Aldrin	0.1	0.5
Heptachlor Epoxide	0.2	0.5
gamma-Chlordane	0.07	0.5
Endosulfan I	0.1	0.5
alpha-Chlordane	0.04	0.5
Dieldrin	0.06	0.5
4,4'-DDE	0.1	0.5
Endrin	0.05	0.5
Endosulfan II	0.06	0.5
4-4'-DDD	0.05	0.5
Endrin Aldehyde	0.04	0.5
Endosulfan Sulfate	0.13	0.5
4,4'-DDT	0.047	0.5
Endrin Ketone	0.03	0.5
Methoxychlor	0.17	0.5
NOAA List		
Hexachlorobenzene	0.1	0.5
Chlorpyrifos	*	0.5
Oxychlordane	*	0.5
2,4'-DDE	0.05	0.5
trans-Nonachlor	*	0.5
2,4'-DDD	0.06	0.5
cis-Nonachlor	*	0.5
2,4'-DDT	0.1	0.5
Mirex	*	0.5

* Analyte typically not requested in water matrix. Call laboratory for further information.

TABLE 5

PCB Aroclors
Gas Chromatography (GC), EPA Method 8082
Method Detection Limits (MDLs) & Method Reporting Limits (MRLs)

	(SPE extraction) Water (µg/L)		Soil/Sediment (µg/Kg) (Dry Wt. Basis)		Tissue (µg/Kg) (Wet Wt. Basis)	
Analyte	**MDL**	**MRL**	**MDL**	**MRL**	**MDL**	**MRL**
Aroclor 1016	0.02	0.2	10	100	2	10
Aroclor 1221	0.04	0.4	6	200	3	20
Aroclor 1232	0.06	0.2	10	100	2	10
Aroclor 1242	0.08	0.2	9	100	4	10
Aroclor 1248	0.02	0.2	4	100	1	10
Aroclor 1254	0.03	0.2	4	100	2	10
Aroclor 1260	0.01	0.2	12	100	5	10
Aroclor 1262	0.07	0.2	5	100	4	10
Aroclor 1268	0.09	0.2	3	100	2	10
Ultra Low-Level	(Requires 2-L aliquot for aqueous samples)					
Aroclor 1016	0.003	0.005			2	2
Aroclor 1221	0.003	0.01			2	4
Aroclor 1232	0.003	0.005			2	2
Aroclor 1242	0.003	0.005			2	2
Aroclor 1248	0.003	0.005			2	2
Aroclor 1254	0.003	0.005			2	2
Aroclor 1260	0.003	0.005			2	2
Aroclor 1262	0.003	0.005			2	2
Aroclor 1268	0.003	0.005			2	2
Low-Level	(Requires 1-L aliquot for aqueous samples)					
Aroclor 1016	0.007	0.02	2	10		
Aroclor 1221	0.007	0.04	2	20		
Aroclor 1232	0.007	0.02	2	10		
Aroclor 1242	0.007	0.02	2	10		
Aroclor 1248	0.007	0.02	2	10		
Aroclor 1254	0.007	0.02	2	10		
Aroclor 1260	0.007	0.02	2	10		
Aroclor 1262	0.007	0.02	2	10		
Aroclor 1268	0.007	0.02	2	10		

TABLE 6

PCB Congeners - Gas Chromatography (GC), EPA Method 8082

Method Detection Limits (MDLs) & Method Reporting Limits (MRLs)

Analyte		Water (ng/L)		Soil/Sediment (µg/Kg) (Dry Wt. Basis)		Tissue (µg/Kg) (Wet Wt. Basis)	
		MDL	MRL	MDL	MRL	MDL	MRL
PCB 1	2-Monochlorobiphenyl	2	5	0.3	0.5	0.5	1
PCB 5	2,3-Dichlorobiphenyl	0.8	5	0.06	0.5	0.2	0.5
PCB 8	2,4'-Dichlorobiphenyl	1	5	0.09	0.5	0.1	2
PCB 18	2,2',5-Trichlorobiphenyl	2	5	0.03	0.5	0.1	0.5
PCB 28	2,4,4'-Trichlorobiphenyl	1	5	0.3	0.5	0.3	0.5
PCB 31	2,4',5-Trichlorobiphenyl	2	5	0.07	0.5	0.1	0.5
PCB 33	2',3,4-Trichlorobiphenyl	2	5	0.1	0.5	0.2	0.5
PCB 37	3,4,4'-Trichlorobiphenyl	0.5	5	0.06	0.5	0.1	0.5
PCB 44	2,2',3,5'-Tetrachlorobiphenyl	2	5	0.2	0.5	0.1	0.5
PCB 49	2,2',4,5'-Trichlorobiphenyl	0.5	5	0.05	0.5	0.1	0.5
PCB 52	2,2',5,5'-Tetrachlorobiphenyl	2	5	0.05	0.5	0.08	1
PCB 56	2,3,3',4'-Trichlorobiphenyl	2	5	0.09	0.5	0.08	0.5
PCB 60	2,3,4,4'-Tetrachlorobiphenyl	0.9	5	0.04	0.5	0.2	0.5
PCB 66	2,3',4,4'-Tetrachlorobiphenyl	1	5	0.04	0.5	0.07	0.5
PCB 70	2,3',4',5-Trichlorobiphenyl	1	5	0.04	0.5	0.1	0.5
PCB 74	2,4,4',5-Trichlorobiphenyl	2	5	0.05	0.5	0.3	0.5
PCB 77	3,3',4,4'-Tetrachlorobiphenyl	0.4	5	0.07	0.5	0.09	0.5
PCB 81	3,4,4',5-Tetrachlorobiphenyl	1	5	0.03	0.5	0.08	0.5
PCB 87	2,2',3,4,5'-Pentachlorobiphenyl	1	5	0.03	0.5	0.2	0.5
PCB 90	2,2',3,4',5-Pentachlorobiphenyl	1	5	0.03	0.5	0.1	0.5
PCB 95	2,2',3,5',6-Pentachlorobiphenyl	0.6	5	0.05	0.5	0.3	0.5
PCB 97	2,2',3',4,5-Pentachlorobiphenyl	1	5	0.03	0.5	0.1	0.5
PCB 99	2,2',4,4',5-Pentachlorobiphenyl	2	5	0.03	0.5	0.06	0.5
PCB 101	2,2',4,5,5'-Pentachlorobiphenyl	2	5	0.03	0.5	0.07	0.5
PCB 105	2,3,3',4,4'-Pentachlorobiphenyl	0.3	5	0.04	0.5	0.2	0.5
PCB 110	2,3,3',4',6-Pentachlorobiphenyl	2	5	0.03	0.5	0.1	0.5
PCB 114	2,3,4,4',5-Pentachlorobiphenyl	1	5	0.08	0.5	0.3	0.5
PCB 118	2,3',4,4',5-Pentachlorobiphenyl	1	5	0.04	0.5	0.1	0.5
PCB 119	2,3',4,4',6-Pentachlorobiphenyl	0.7	5	0.06	0.5	0.1	0.5
PCB 123	2',3,4,4',5-Pentachlorobiphenyl	1	5	0.03	0.5	0.1	0.5
PCB 126	3,3',4,4',5-Pentachlorobiphenyl	0.3	5	0.04	0.5	0.1	0.5
PCB 128	2,2',3,3',4,4'-Hexachlorobiphenyl	0.4	5	0.3	0.5	0.09	0.5
PCB 132	2,2',3,3',4,6'-Hexachlorobiphenyl	2	5	0.03	0.5	0.3	0.5
PCB 138	2,2',3,4,4',5'-Hexachlorobiphenyl	1	5	0.03	0.5	0.2	0.5
PCB 141	2,2',3,4,5,5'-Hexachlorobiphenyl	1	5	0.03	0.5	0.1	0.5
PCB 149	2,2',3,4',5',6-Hexachlorobiphenyl	0.5	5	*	0.5	*	0.5
PCB 151	2,2',3,5,5',6-Hexachlorobiphenyl	1	5	0.06	0.5	0.07	0.5
PCB 153	2,2',4,4',5,5'-Hexachlorobiphenyl	0.9	5	0.04	0.5	0.2	0.5
PCB 156	2,3,3',4,4',5-Hexachlorobiphenyl	1	5	0.04	0.5	0.1	0.5
PCB 157	2,3,3',4,4',5'-Hexachlorobiphenyl	2	5	0.04	0.5	0.08	0.5
PCB 158	2,3,3',4,4',6-Hexachlorobiphenyl	0.3	5	0.04	0.5	0.08	0.5
PCB 166	2,3,4,4',5,6,-Hexachlorobiphenyl	0.5	5	0.04	0.5	0.1	0.5
PCB 167	2,3',4,4',5,5'-Hexachlorobiphenyl	2	5	0.03	0.5	0.2	0.5
PCB 168	2,3',4,4',5',6-Hexachlorobiphenyl	0.6	5	0.04	0.5	0.08	0.5
PCB 169	3,3',4,4',5,5'-Hexachlorobiphenyl	0.3	5	0.03	0.5	0.09	0.5
PCB 170	2,2',3,3',4,4',5-Heptachlorobiphenyl	0.4	5	0.03	0.5	0.08	0.5
PCB 174	2,2',3,3',4,5,6'-Heptachlorobiphenyl	0.4	5	0.2	0.5	0.06	0.5
PCB 177	2,2',3,3',4',5,6-Heptachlorobiphenyl	1	5	0.09	0.5	0.3	0.5
PCB 180	2,2',3,4,4',5,5'-Heptachlorobiphenyl	1	5	0.03	0.5	0.1	0.5
PCB 183	2,2',3,4,4',5',6-Heptachlorobiphenyl	1	5	0.03	0.5	0.2	0.5
PCB 184	2,2',3,4,4',6,6'-Heptachlorobiphenyl	2	5	0.05	0.5	0.08	0.5
PCB 187	2,2',3,4',5,5',6-Heptachlorobiphenyl	2	5	0.04	0.5	0.2	0.5
PCB 189	2,3,3',4,4',5,5'-Heptachlorobiphenyl	1	5	0.03	0.5	0.09	0.5
PCB 194	2,2',3,3',4,4',5,5'-Octachlorobiphenyl	1	5	0.03	0.5	0.3	0.5
PCB 195	2,2',3,3',4,4',5,6-Octachlorobiphenyl	1	5	0.04	0.5	0.1	0.5
PCB 201	2,2',3,3',4,5',6'c-Octachlorobiphenyl	2	5	0.03	0.5	0.3	0.5
PCB 203	2,2',3,4,4',5,5',6-Octachlorobiphenyl	1	5	0.03	0.5	0.2	0.5
PCB 206	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	2	5	0.06	0.5	0.08	0.5
PCB 209	2,2',3,3',4,4',5,5',6,6'Decachlorobiphenyl	2	5	0.05	0.5	0.1	0.5

* Please contact Laboratory for latest limits

TABLE 7

PCB Coplanar Congeners - HRGC/HRMS, EPA Method 1668A

PCB Congener World Health Organization (WHO) List

Method Reporting Limits (MRLs)*

Analyte		TEF**	Water (pg/L) MRL	Soil/Sediment (ng/Kg) (Dry Wt. Basis) MRL	Tissue (ng/Kg) (Wet Wt. MRL
PCB 77	3,3',4,4'-Tetrachlorobiphenyl	0.0001	500	50	50
PCB 81	3,4,4',5-Tetrachlorobiphenyl	0.0001	500	50	50
PCB 105	2,3,3',4,4'-Pentachlorobiphenyl	0.0001	200	20	50
PCB 114	2,3,4,4',5-Pentachlorobiphenyl	0.0005	500	50	50
PCB 118	2,3',4,4',5-Pentachlorobiphenyl	0.0001	500	50	50
PCB 123	2',3,4,4',5-Pentachlorobiphenyl	0.0001	500	50	50
PCB 126	3,3',4,4',5-Pentachlorobiphenyl	0.1	500	50	50
PCB 156	2,3,3',4,4',5-Hexachlorobiphenyl	0.0005	500	50	50
PCB 157	2,3,3',4,4',5'-Hexachlorobiphenyl	0.0005	500	50	50
PCB 167	2,3',4,4',5,5'-Hexachlorobiphenyl	0.00001	500	50	50
PCB 169	3,3',4,4',5,5'-Hexachlorobiphenyl	0.01	500	50	50
PCB 189	2,3,3',4,4',5,5'-Heptachlorobiphenyl	0.0001	500	50	50

* Please contact Laboratory for latest limits, RLs can be adjusted to meet project requirements.
** Toxicity Equivalency Factor

TABLE 8

Organotins

Method Detection Limits (MDLs) & Method Reporting Limits (MRLs)

	Water/Porewater (*u* g/L) M. A. Unger, et al. (GC/FPD)		Soil/Sediment (*u* g/Kg) (Dry Wt. Basis) C. A. Krone, et al. (GC/FPD)		Tissue (*u* g/Kg) (Wet Wt. Basis) M. O. Stallard, et al. (GC/FPD)	
Analyte	**MDL**	**MRL**	**MDL**	**MRL**	**MDL**	**MRL**
Tetra-n-butyltin	0.003	0.05	0.1	1	0.4	1
Tri-n-butyltin	0.007	0.02	0.2	1	0.3	1
Di-n-butyltin	0.005	0.05	0.04	1	0.4	1
n-butyltin	0.005	0.05	0.07	1	0.4	1

TABLE 9

EPA Method 200.8/6020

Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

Method Detection Limits (MDLs) & Method Reporting Limits (MRLs)

	Water (µg/L)		Soil/Sediment (mg/Kg) (Dry Wt. Basis)		Tissue (mg/Kg) (Wet Wt. Basis)	
Analyte	**MDL**	**MRL**	**MDL**	**MRL**	**MDL**	**MRL**
Aluminum	2	2	2	2	0.06	0.4
Antimony	0.02	0.05	0.02	0.05	0.002	0.01
Arsenic	0.2	0.5	0.07	0.5	0.006	0.1
Barium	0.03	0.05	0.03	0.05	0.004	0.01
Beryllium	0.007	0.02	0.006	0.02	0.002	0.004
Cadmium	0.02	0.02	0.07	0.05	0.002	0.004
Chromium	0.06	0.2	0.04	0.2	0.01	0.1
Cobalt	0.01	0.02	0.01	0.02	0.0006	0.004
Copper	0.03	0.1	0.02	0.1	0.02	0.02
Lead	0.009	0.02	0.02	0.05	0.002	0.004
Manganese	0.02	0.05	0.04	0.05	0.001	0.01
Molybdenum	0.02	0.05	0.008	0.05	0.001	0.01
Nickel	0.2	0.2	0.04	0.2	0.006	0.04
Selenium	0.6	1	0.2	1	0.08	0.2
Silver	0.009	0.02	0.003	0.02	0.0008	0.004
Thallium	0.004	0.02	0.002	0.02	0.0004	0.004
Uranium	0.006	0.02	0.004	0.02	0.0004	0.004
Vanadium	0.03	0.2	0.03	0.2	0.1	0.2
Zinc	0.3	0.5	0.2	0.5	0.02	0.1

*Chromium and Vandium in tissue are analyzed by ICP-OES, Selenium is analyzed by GFAAS.

Lower limits are available for Selenium when using Hydride AAS.

EPA Method 1631M - Mercury by Atomic Fluorescence MDLs and MRLs

	Water (µg/L)		Sediment (mg/Kg)	
	MDL	**MRL**	**MDL**	**MRL**
Mercury	0.00006	0.001	0.0002	0.002

EPA Method 7471A - Mercury by CVAAS MDLs and MRLs *

	Sediment (mg/Kg) (Dry Wt. Basis)		Tissue (mg/Kg) (Wet Basis)	
	MDL	**MRL**	**MDL**	**MRL**
Mercury	0.008	0.02	0.002	0.004

*Lower detection limit for Hg in tissue is available. Call for specifications.

TABLE 10

Reductive Precipitation

Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)
Method Reporting Levels (MRLs)

Analyte	Seawater µg/L MDL	MRL
Arsenic	0.02	0.5
Beryllium	0.0008	0.02
Cadmium	0.003	0.02
Chromium	0.02	0.2
Cobalt	0.002	0.02
Copper	0.008	0.1
Lead	0.009	0.02
Nickel	0.02	0.2
Silver	0.005	0.02
Thallium	0.0006	0.02
Zinc	0.02	0.5

TABLE 11

Regulated Dioxin and Furan Isomers

HRGC/HRMS

SW 846 Method 8290

Dioxins	Reporting Limits* Water (pg/L)	Reporting Limits* Solids (ng/Kg)
2,3,7,8-TCDD	10	1
1,2,3,7,8-PeCDD	25	2.5
1,2,3,4,7,8-HxCDD	25	2.5
1,2,3,6,7,8-HxCDD	25	2.5
1,2,3,7,8,9-HxCDD	25	2.5
1,2,3,4,6,7,8-HpCDD	25	2.5
OCDD	50	5

Furans	Reporting Limits* Water (pg/L)	Reporting Limits* Solids (ng/Kg)
2,3,7,8-TCDF	10	1
1,2,3,7,8-PeCDF	25	2.5
2,3,4,7,8-PeCDF	25	2.5
1,2,3,4,7,8-HxCDF	25	2.5
1,2,3,6,7,8-HxCDF	25	2.5
2,3,4,6,7,8-HxCDF	25	2.5
1,2,3,7,8,9-HxCDF	25	2.5
1,2,3,4,6,7,8-HpCDF	25	2.5
1,2,3,4,7,8,9-HpCDF	25	2.5
OCDF	50	5

* Actual reporting limits vary from sample to sample.

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Appendix B

Test Procedures and SOPs for Bioassays

SOP No. 4050	Rev. No.: 1.0	Date: May 7, 2010
***Leptocheirus plumulosus* 28d Sediment Survival, Growth and Reproduction**		**Reference Method: EPA 600/R-01/020**

1.0 PURPOSE AND APPLICABILITY

This procedure establishes a standard method for conducting whole sediment toxicity tests using the amphipod Leptocheirus plumulosus. Endpoints assessed using this SOP include survival (number of live organisms at the end of the exposure period), growth (average dry-weight/surviving organism) and reproduction (number of offspring per living adult).

2.0 REFERENCES

References listed in this section are incorporated into this SOP.

US EPA. 2001. Method for Assessing the Chronic Toxicity of Marine and Estuarine Sediment-associated Contaminants with the Amphipod Leptocheirus plumulosus. EPA/600/R-01/020.

SOP 5006: Calibration, Operation and Maintenance of the Orion Model 210A pH Meter

SOP 5007: Calibration, Operation and Maintenance of the Orion Model 410A pH Meter

SOP 5008: Calibration, Operation, and Maintenance of the Orion 3 Star pH Meter

SOP 5002: Calibration, Operation and Maintenance of the YSI Model 55 Dissolved Oxygen and Temperature System

SOP 5003; Calibration, Operation and Maintenance of the YSI Model 30 Handheld Salinity, Conductivity and Temperature System

SOP 5004; Calibration, Operation and Maintenance of the YSI Model 3100 Salinity, Conductivity and Temperature System

SOP 5016; Ammonia Determination with the Orion Model 720A pH/ISE Meter and Orion Model 95-12 Ammonia Electrode: Calibration, Operation and Maintenance

3.0 DEFINITIONS

whole sediment - sediment and associated porewater that have had minimal manipulation

overlying water - water placed over sediment in test chamber during test

control sediment - whole sediment which has been demonstrated to be suitable for use as a control medium. Control sediment should be capable of supporting attainment of test acceptability criteria in a high percentage of tests.

dead - Test organisms are "dead" if they exhibit (1) no movement of appendages and (2) no reaction to gentle prodding.

interferences - characteristics of sediment or sediment test system that could affect test organism survival, aside from those related to sediment-associated contaminants

4.0 HEALTH AND SAFETY CONSIDERATIONS

Whole sediments submitted by project sponsors for toxicity characterization are potentially hazardous – handle with appropriate care. Study Director provides additional hazard warnings and safety information for handling sediments.

Wear standard laboratory personal safety equipment (gloves, lab coat, and safety glasses) when preparing or handling whole sediments.

5.0 QUALITY ASSURANCE AND PLANNING CONSIDERATIONS

Variations from this procedure are not anticipated or encouraged. Justify study-specific changes in a study protocol, work plan or test notebook, and evaluate (in writing) with respect to potential effects on this procedure.

6.0 RESPONSIBILITIES

Special Projects Director

- specifies this procedure.
- advises laboratory staff on H&S considerations that apply to test sediments.
- notifies laboratory staff of any special testing instructions.

NOTE: The latter two responsibilities are addressed in study protocol and/or test notebooks, and are discussed with key members of study team before study initiation.

Special Projects Manager (or other designated staff member)assures that assigned personnel are fully trained to perform this procedure.

Laboratory Technicians follow this procedure as specified.

7.0 TRAINING/QUALIFICATIONS

No specific training or qualifications, other than documented training to the requirements of this SOP, are required; training records for all personnel assigned to perform this procedure are current.

8.0 REQUIRED MATERIALS

- site and reference sediments
- control sediment
- US Standard Sieves, 0.25mm, 0.6mm stainless steel
- round, opaque Nalgene bowls
- laboratory-prepared seawater, 5 ± 2ppt or 20 ± 2ppt salinity (project specific)
- pH meter
- dissolved oxygen meter
- stainless steel forceps
- water diffuser
- TetraMin® slurry.
- 4L pitcher
- balance
- dissecting probe
- dissecting microscope
- ammonia ion probe & meter
- 1-L glass jars
- aeration system
- 40mL plastic disposable cups
- plastic tray with holding cups
- 1X5cm, 450µm Nitex® mesh loading net, or wide-bore dropping pipette
- test organisms, 2-4 mm *L. plumulosus*
- salinity meter
- thermometer
- centrifuge and tubes
- 8% sugar-formalin solution (mix 120g sucrose and 80 mL formalin; bring to 1-L)
- 5mL disposable serological glass pipette and pipette device
- drying oven
- pre-weighed aluminum weigh boats

9.0 SAMPLE COLLECTION PRESERVATION AND STORAGE

Handle, preserve and store samples to minimize changes in composition and avoid contamination. Store sediments in darkened cooler at 1-6°C until use.

Elapsed time between sample collection and analysis should be as short as possible; for biological testing, use samples within two weeks of collection, but they may be stored up to six weeks.

10.0 METHOD

10.1 Sample Manipulation

Store sediments in darkened cooler at 1-6°C, prior to use.

10.1.1 **Homogenization**

Samples tend to settle during shipment. As a result, water may collect above the sediment. This water should not be discarded, but should be mixed back into the sediment during homogenization. Homogenize the sample by manually mixing the sediment and associated water with a large polyethylene or Teflon spoon.

Homogenize sediments directly in the original container or, if multiple aliquots are provided, in a non-contaminating vessel such as a polyethylene mixing bowl. Pick stones, sticks, large organisms, or other debris from the sediments with stainless steel forceps.

10.1.2 **Pore-Water Extraction**

After homogenization, for each sediment sample to be tested, put a 25mL aliquot of sediment into a 50mL centrifuge tube. Spin the sediment in the centrifuge at ~ 850 xG (2000 rpm for the HNS benchtop centrifuge) for at least 20 minutes to separate pore-water from the sediment. Repeat as many times as necessary to get 50mL of pore-water from each sediment sample. Measure ammonia in the pore-water samples by ion probe as well as pH and salinity and record results.

10.1.3 **Do not sieve** the test sediments unless there is a concern about indigenous organisms that may influence the response of the test organism. Prepare approximately 0.2L sediment per replicate. Return sediments to storage area in air-tight containers. If determined necessary by the Study Director, press-sieve test and reference sediments through a stainless steel screen before use in tests. Sieve size is project-specific and will be determined by the Study Director. Minimize sediment handling and manipulation; sieve samples as close as possible to test day to avoid changes in chemical bioavailability. Sieve only the amount of sediment needed for testing. Make note in Special Projects notebook of which sediments were sieved and sediment condition prior to sieving.

10.2 Control Medium

Use clean sand or native sediment as control medium. Press-sieve sediment before use, using 1.0mm stainless steel sieve.

10.3 Experiment Design

Test is 28d static renewal.

10.4 Test Vessels

Test vessels are 1-L wide-mouth glass jars.

10.5 Test Organisms

L. plumulosus are small, laterally compressed amphipods. Use 2-4 mm amphipods for testing.

Order *L. plumulosus* from commercial vendor: at least 20 organisms per replicate in advance for arrival on test initiation day (Day 0). If organisms arrive sooner, place in covered glass aquarium; provide 16:8D hours photoperiod and gentle aeration.

10.6 Test Water

Water overlying sediments in test containers is lab prepared synthetic seawater. Overlying water in each test vessel is renewed 3 x week (48h intervals); begin water renewals on day 0.

10.7 Test Initiation

10.7.1 Day Minus 1 (-1)

1) Distribute well homogenized test and control sediments into 1-L glass jars to depth of about 2cm (~175mL).
2) Settle sediment by tapping bottom of test chamber gently on flat surface.
3) Measure and record water quality parameters (pH, DO, salinity and temperature) of fresh seawater to be used as overlying water.
4) Using diffuser, pour ~775mL of laboratory-prepared seawater over test material. Clean diffuser between treatments.
5) Place test vessels in 25 ± 2° C, 16L:8D light area and provide gentle aeration (<100 bubbles/min). Allow sediment to settle overnight.

10.7.2 Day 0

1) Measure and record old and new water quality parameters of test (see Section 10.8.1).
2) Carefully pour or siphon ~80% of overlying water from each test vessel.
3) Fill each chamber with fresh seawater. To minimize disturbance of sediment, use diffuser over sediment and allow water to discharge directly onto diffuser.
4) When test organisms arrive, provide aeration and allow organisms to acclimate to test temperature in original shipping container. After an hour of acclimation, pour organisms from original shipping container into a nalgene bowl and put on aeration.

10.7.3 **Day 0 - test organism loading**

1) Place 40mL plastic cups (2 per test replicate) on plastic tray equipped with holding cups. Pour ~30mL seawater into each cup.
2) Select healthy, active, non-gravid sub-adults of uniform size and use small fine-mesh Nitex net or wide-bore dropping pipette to load 10 into each 40mL cup. Load two cups for each test replicate.
3) Have second technician confirm that correct number of organisms are loaded.
4) When organisms are loaded, transfer carefully into test vessels by pouring entire contents of randomly selected loading cup directly into each vessel.
5) Push organisms caught in surface tension of water gently into water column, using blunt end of glass rod.
6) At end of day, inspect all test vessels; remove and replace any organisms that have returned to water surface.
7) Restore gentle aeration (<100 bubbles/minute).

10.8 Test Maintenance

Maintain test vessels at 25 ± 2°C with 16L:8D hours photoperiod at illuminance ~500-1000fc. Provide constant aeration (<100 bubbles/min).

10.8.1 **Water quality measurments**

Measure and document water quality parameters (pH, D.O., salinity, total ammonia and temperature) of overlying water daily and (pH, D.O., salinity, temperature) of renewal water on renewal days.

10.8.2 **Water renewals**

Renew overlying water in each test vessel every 48h. Carefully pour or siphon ~80% of overlying water from each test vessel. Set aside a 250mL aliquot for new water quality measurements (pH, DO, salinity and temperature). Fill each chamber with fresh seawater to minimize disturbance of sediment, use diffuser over sediment and allow water to discharge directly onto diffuser.

10.8.3 **Test Feeding**

Feed test vessels 1mL TetraMin® slurry after water renewal. (TetraMin® is fed at a rate of 20mg per test vessel days 0-13 and 40mg per test vessel days 14-28.) See SOP # 3001 for slurry preparation instructions.

10.9 Daily test observations

Inspect all test vessels on Days 1-28 and record amphipod behavior on data sheet as follows:

E = **Emergent**, organisms present in water column, on sediment surface or water surface, but not burrowing. Include number of organisms exhibiting this behavior (*e.g.*, 3-E).

D = **Dead**, organisms do not respond to gentle prodding and there is no movement of appendages. Remove and discard "dead" organisms. Include number of organisms exhibiting this behavior (*e.g.*, 3-D).

✓= **All organisms burrowing**, no organisms visible.

10.10 Test Termination

10.10.1 Survival

1) On Day 28, measure and document water quality parameters (ph, DO, salinity, total ammonia, and temperature) for each test treatment.
2) Arrange 40mL labled plastic cups (one per test replicate) on tray equipped with holding cups. Pour ~30mL of seawater into each cup (test organisms are transferred to cups for survival counts).
3) Stack a 0.6mm sieve over a clean bucket or container to capture wash through. Working with one replicate at a time, pour approximately half overlying water onto sieve. Swirl remaining contents of the test vessel gently to allow sediment to suspend into overlying water.
4) Pour approximately half remaining contents onto sieve.
5) Use spray nozzle to re-fill test vessel gently; swirl vessel and pour remaining sediment over screen. Rinse test vessel onto sieve.
6) Rinse sieve quickly but gently with tap water to remove sediment particles, and place sieve in Nalgene bowl filled with seawater. Use small fine-mesh loading net or wide-bore dropping pipette to transfer any organisms that emerge to water surface into 40mL disposable plastic cups containing about 10mL of 8% formalin solution.
7) With screen still in bowl, spin screen and tap gently to induce amphipods to emerge; transfer into plastic cups. **NOTE:** Work slowly and gently through remaining sediment until confident that all organisms have been recovered.
8) Count and record number of surviving organisms in 40mL plastic cups. Have second technician confirm survival counts; if counts do not agree, have third technician make counts. Record all counts on QAU form 3570. Set aside surviving organisms for weight determination.

10.10.2 **Growth**

1) Draw off sugar-formalin solution (See section 10.9.1 #6) with a pipette and rinse organisms twice with ~10mL aliquots of de-ionized water.
2) Transfer organisms from each replicate to tarred, labeled weigh-boats and dry the organisms at 60°C for 24h.
3) After drying is complete, cool organisms to room temperature in a desiccator and weigh to the nearest 0.01mg. Record measurements on QAU form 3570.

10.10.3 **Reproduction**

1) Pour contents remaining in the capture bucket (See section 10.9.1 #3) through a 0.25mm screen and use spray nozzle to rinse sieve gently and quickly.
2) Rinse live neonates captured on the 0.25mm screen into a shallow dish and count them using a dissecting microscope. Record on QAU form 3570.

11.0 INTERFERENCES

1) Characteristics of sediment that may affect test organism survival, independent of contaminant concentration.
2) Changes in chemical bioavailability as function of sediment manipulation or storage.
3) Presence of indigenous organisms.

12.0 QUALITY CONTROL CHECKS AND ACCEPTANCE CRITERIA

Calibrate all measuring equipment used (pH, meters, ammonia meter, thermometers) per established procedures.

Acceptance criteria for control group:

- survival ≥80%, with no single replicate having ≤ 60% survival.
- measurable growth and reproduction in all replicates
- reference toxicant LC_{50}'s within control limits (±2sd from mean).

Reference toxicant evaluations: 96h water-only tests with cadmium as toxicant.

13.0 CORRECTIVE ACTIONS AND CONTINGENCIES FOR OUT-OF-CONTROL DATA

Re-run any toxicity test which does not meet minimum acceptance criteria for control survival (see Section 12.0).

14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Unless otherwise directed by project sponsor, place remaining test material (used and unused) in sealed HDPE buckets and discard in waste collection container.

15.0 DOCUMENTATION

Document water quality parameters and survival counts on QAU form #3570. Document changes to protocol in test notebook. Archive original data at PBS&J Environmental Toxicology Laboratory.

TABLE 9.1 TEST CONDITION SUMMARY: 28D SEDIMENT TOXICITY TEST WITH *L. plumulosus*	
TEST TYPE:	whole sediment toxicity test; static
TEMPERATURE:	25 ± 2°C
SALINITY:	5 ± 2‰ 20 ± 2‰
LIGHT QUALITY:	Wide-spectrum fluorescent lights
ILLUMINANCE:	500-1000 lux
PHOTOPERIOD:	16L:8D
TEST CHAMBER:	1-L glass beaker or jar with 10cm I.D.
SEDIMENT VOLUME:	175mL (2cm)
OVERLYING WATER VOLUME:	775mL
RENEWAL OF OVERLYING WATER:	3 x per week; siphon and replace ~80% overlying water
SIZE AND LIFE STAGE OF AMPHIPODS:	2-4mm (use specimens which pass through a 0.6mm sieve and are retained on a 0.25mm sieve)
NUMBER OF ORGANISMS:	20 per test chamber
NUMBER OF REPLICATES:	Depends on test objective -- minimum 5
FEEDING:	3 x per week after renewal; days 0-13, 20mg TetraMin® per test vessel; Days 14-28, 40mg TetraMin® per test vessel
AERATION:	Aerate water in each test chamber overnight before start of test, and throughout the test, at rate that maintains ≥90% saturation of dissolved oxygen concentration
OVERLYING WATER:	Clean seawater, natural or reconstituted water
OVERLYING WATER QUALITY:	Temperature, pH, total ammonia, salinity, and DO of overlying water daily. Temperature, pH, salinity and DO of renewal water at renewal. Salinity, ammonia and pH of pore water
TEST DURATION:	28d
ENDPOINTS:	Survival, reproduction and growth
TEST ACCEPTABILITY:	Minimum mean control survival of 80%; growth and reproduction measurable in all control replicates and satisfaction of performance-based criteria outlined in Table 11.3 of EPA 600/R-01/020.

SOP No. 4049	Rev. No.: 1.0	Date: May 6, 2010
***Neanthes arenaceodentata* 28d Survival & Growth Test**		**Reference Method: ASTM E 1611**

1.0 PURPOSE AND APPLICABILITY

This procedure establishes a standard method for conducting a 28-day sediment toxicity test with the polychaete *Neanthes arenaceodentata*. Endpoints assessed using this SOP included survival (number of live organisms at the end of the exposure period) and growth (average dry-weight/surviving organism).

This procedure is applicable where sediment assessment requires a more sensitive endpoint(s) than may be achieved with a shorter exposure duration (e.g. 10d test).

2.0 REFERENCES

References listed in this section are incorporated into this SOP.

ASTM International. *Standard Guide for Conducting Sediment Toxicity Tests with Polychaetous Annelids.* E 1611-00.

SOP 5002: Calibration, Operation and Maintenance of the YSI Model 55 Dissolved Oxygen and Temperature System

SOP 5003: Calibration, Operation and Maintenance of the YSI Model 30 Handheld Salinity, Conductivity and Temperature System

SOP 5004: Calibration, Operation and Maintenance of the YSI Model 3100 Salinity, Conductivity and Temperature System

SOP 5006: Calibration, Operation and Maintenance of the Orion Model 210A pH Meter

SOP 5007: Calibration, Operation and Maintenance of the Orion Model 410A pH Meter

SOP 5008: Calibration, Operation, and Maintenance of the Orion 3 Star pH Meter

SOP 5016: Ammonia Determination with the Orion Model 720A pH/ISE Meter and Orion Model 95-12 Ammonia Electrode: Calibration, Operation and Maintenance

3.0 DEFINITIONS

whole sediment - sediment and associated pore water that have had minimal manipulation

overlying water - water placed over sediment in test chamber during test

control sediment - sediment essentially free of contaminants, used routinely to assess acceptability of a test. Control sediment may be sediment from which the test organisms are collected or clean beach sand.

reference sediment - whole sediment collected near area of concern, used to assess sediment conditions exclusive of materials of interest. Reference sediment may be used as indicator of localized sediment conditions exclusive of specific pollutant input of concern.

dead - test organisms are "dead" if they exhibit (1) no movement, and (2) no reaction to gentle prodding

interferences - characteristics of a sediment or sediment test system that could affect test organism survival, aside from those related to sediment- associated contaminants.

4.0 HEALTH AND SAFETY CONSIDERATIONS

Whole sediments submitted by project sponsors for toxicity characterization are potentially hazardous -- handle with appropriate care. Study Director provides additional hazard warnings and safety information for handling sediments.

Wear standard laboratory personal safety equipment (gloves, lab coat, and safety glasses) when preparing or handling whole sediments.

5.0 QUALITY ASSURANCE AND PLANNING CONSIDERATIONS

Variations from this procedure are not anticipated or encouraged. Justify study-specific amendments in study protocol, work plan, or test notebook, and evaluate (in writing) with respect to potential effects on this procedure.

6.0 RESPONSIBILITIES

Special Projects Director

- specifies this procedure.
- advises laboratory staff regarding H&S considerations that apply to test sediments.
- notifies laboratory staff of special testing instructions.

NOTE: The latter two responsibilities are addressed in study protocol and/or test notebooks, and are discussed with key members of study team prior to study initiation.

Special Projects Manager (or other designated staff member) assures that assigned personnel are fully trained to perform this procedure.

Laboratory Technicians follow this procedure as specified.

7.0 TRAINING/QUALIFICATIONS

No specific training or qualifications, other than documented training to the requirements of this SOP, are required; training records for all personnel assigned to perform this procedure are current.

8.0 REQUIRED MATERIALS

- test sediments
- reference sediment
- control medium (beach sand or other)
- 500µm and 1.0mm stainless steel sieve
- laboratory prepared seawater 25-32ppt (project specific)
- pH meter
- dissolved oxygen meter
- salinity meter
- ammonia probe and meter
- turkey baster
- water diffuser
- TetraMarin® flake/Alfalfa mixture; 4 mg dry-solid per mL suspension.
- 4L pitcher
- test vessels (1L glass jars)
- aeration system
- test organisms (*Neanthes arenaceodentata,* juvenile 2-3 weeks old)
- 40mL plastic disposable cups
- balance
- dissecting probe
- drying oven
- pre-weighed 1x1.5cm aluminum pans
- thermometer
- centrifuge and tubes
- stainless steel forceps
- 8% sugar-formalin solution (mix 120g sucrose and 80mL formalin; bring to 1-L)
- 10mL disposable serological glass pipette and pipette device.

9.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

Handle, preserve and store samples to minimize changes in composition and avoid contamination. Place sediments in non-contaminating containers (high- density polyethylene, Teflon, etc.) and seal tightly with minimum head space. Store in darkened cooler at 1-6°C until use.

Elapsed time between sample collection and analysis should be as short as possible; for biological testing, use samples within two weeks of collection, but they may be stored up to six weeks.

10.0 METHOD

10.1 Sediment Samples

Store sediments in darkened cooler at 1-6°C, prior to use.

10.1.1 **Homogenization**
Samples tend to settle during shipment. As a result, water may collect above the sediment. This water should not be discarded, but should be re-mixed into the sediment during homogenization. Homogenize the sample by manually mixing the sediment and associated water with a large polyethylene or Teflon spoon. Homogenize sediments directly in the original container or, if multiple aliquots are provided, in a non-contaminating vessel such as a polyethylene or stainless steel mixing bowl. Pick stones, sticks, large organisms, or other debris from the sediments with stainless steel forceps.

10.1.2 **Pore-Water Extraction**
After homogenization, for each sediment sample to be tested, put a 25mL aliquot of sediment into a 50mL centrifuge tube. Spin the sediment in the centrifuge at ~ 850 xG (2000 rpm for the HNS benchtop centrifuge) for at least 20 minutes to separate pore-water from the sediment. Repeat as many times as necessary to get 50mL of pore-water from each sediment sample. Measure ammonia, pH, temperature, D.O., and salinity of the pore-water samples and record results (QAU #7420b).

10.1.3 **Do not sieve** the test sediments unless there is a concern about indigenous organisms that may influence the response of the test organism. Prepare approximately 1 gal sediment per replicate. Return sediments to storage area in air-tight containers. If determined necessary by the Study Director, press-sieve test and reference sediments through a stainless steel screen before use in tests. Sieve size is project-specific and will be determined by the Study Director.

Minimize sediment handling and manipulation; homogenize samples as close as possible to test day to avoid changes in chemical bioavailability. Process only the amount of sediment needed for testing. Make note in Special Projects notebook of which sediments were sieved and sediment condition prior to sieving.

10.2 Control Medium

Sieve control medium before use with1.0 mm stainless steel sieve. If beach sand is used, sieve as soon after collection as possible.

10.3 Experiment Design

Test is 28d static renewal with 5 replicates per treatment.

10.4 Test Vessels

Test vessels are 1L glass jars.

10.5 Test Organisms

Use 2-3 week old juvenile *N. arenaceodentata*; 5 organisms per replicate. Order from commercial vendor in advance to arrive on test initiation day (Day 0).

10.6 Test Water

Water overlying sediments in test containers is lab prepared synthetic seawater. Overlying water in each test vessel is renewed one time per day; begin water renewals at day 0 and continue through day 28.

Prior to each renewal, pour 3.3L fresh seawater into one 4L measuring pitcher for for each set of replicate test jars. Using a disposable serological glass pipette, add 5mL of TetraMarin®/Alfalfa suspension (SOP #3001 modified) to each pitcher. Stir contents of the pitcher thoroughly with the pipette.

10.7 Test Initiation

10.7.1 Day Minus 1 (-1)

1) Distribute well-homogenized (sieved, if required) test, reference sediment, and control sand into 1-L glass jars to depth of about 2cm (~175mL).
2) Settle sediment by tapping bottom of test chamber gently on flat surface.
3) Measure and record ph, DO, salinity and temperature of fresh seawater to be used as overlying water (see section 10.8.1).
4) Fill each chamber with fresh seawater. To minimize disturbance to sediment, use diffuser over sediment and allow water to discharge directly onto diffuser.
5) Provide moderate aeration, cover test chambers and allow test sediment to settle overnight.

10.7.2 **Day 0 - Organism Loading**

1) Measure and record old and new water quality parameters of test (see Section 10.8.1 and 10.8.2).
2) Carefully pour or siphon ~80% of overlying water from each test vessel.
3) Fill each chamber with fresh seawater. To minimize disturbance of sediment, use diffuser over sediment and allow water to discharge directly onto diffuser.
4) Set aside 40 mL plastic cups (as many as there are jars).
5) When test organisms arrive, document condition of organisms (QAU Form #6109) and and transfer to 40mL cups; allow organisms to acclimate to test temperature.
6) Load 5 polychaetes into each cup – select healthy organisms, avoid those that are discolored of have skin abscesses.
7) When organisms are loaded, transfer carefully into test vessels by pouring entire contents of a randomly selected loading cup directly into each test vessel.
8) Observe test vessels to make sure all organisms are submerged below the water surface and begin burrowing into the sediment. Replace specimens that do not burrow within two hours.

10.7.3 **Day 0 - Archive Organisms**

1) Collect another group of organisms (archive group) containing an equal number of replicates and organisms per replicate as the other treatments. Siphon most of the water from the archive organism cups and replace with ~10 mL of 8% sugar-formalin solution.
2) After ~20 minutes, draw off the sugar-formalin solution with a pipette and rinse organisms twice with ~10 mL aliquots of de-ionized water. Transfer the archive organisms to tarred weigh boats and dry organisms at 50 ± 2° for 24 hours.
3) After drying, cool weigh-boats to room temperature in a desiccator and weigh to the nearest 0.01 mg. Record measurements on QAU form 3560.

10.8 Test Maintenance

Maintain test vessels at 20 ± 1°C (per study work plan), with 16L:8D photoperiod at illuminance ~50-100fc and constant moderate aeration. Test vessels get fed once per day during water renewal a tetramarin/alfalfa suspension.

10.8.1 **1 X day:** Measure water quality of overlying water and make observations.

1) Inspect test vessels for adequate aeration.
2) Remove dead organisms by pipette, discard appropriately (see Section 3.0 for "dead" criteria) and record observations on QAU form 3560 under "observations".

3) Collect overlying water using a turkey baster; suction ~40-50 mL from each replicate within a site and composite into a 250 mL pre-labeled cup for that site; do this for all sites, control, and reference.
4) Measure water quality parameters (pH, D.O., salinity, temperature, ammonia) and document (QAU 7420a, 3560).

10.8.2 **1 X day:** Renew overlying water.

1) Use turkey baster to siphon and discard ~80% of overlying water from each test vessel.
2) Measure and document water quality parameters (pH, D.O., salinity, and temperature) for new water.
3) Refill jars with fresh test water (see section 10.6), using diffuser to minimize disturbance of test material.
4) Restore aeration and cover test vessels.

10.9 Daily test observations

Inspect all test vessels on Days 0-10 and record amphipod behavior on data sheet as follows:

E = **Emergent**, organisms present in water column, on sediment surface or water surface, but not burrowing. Include number of organisms exhibiting this behavior (*e.g.*, 3-E).

D = **Dead**, organisms do not respond to gentle prodding and there is no movement of appendages. Remove and discard "dead" organisms. Include number of organisms exhibiting this behavior (*e.g.*, 3-D).

✓= **All organisms burrowing**, no organisms visible.

10.10 Test Termination

1) On Day 28, measure and document water quality parameters as described in section 10.8.1. Working with one replicate at a time, pour contents of each test vessel onto 500µm stainless steel sieve. Rinse gently with de-ionized water or tap water to wash away sediment.
2) Place sieve in transparent bowl containing fresh seawater. Count and record number of surviving organisms on QAU form 3560; transfer surviving organisms to a labeled, 40-mL cup containing about 10 mL of 8% sugar-formalin solution.
3) After 20 minutes, draw off sugar-formalin solution with a pipette, and rinse organisms twice with ~10 mL aliquots of de-ionized water.

4) Transfer organisms to tarred, weigh-boat and dry the samples at 50 ± 2°C for 24h.
5) Cool samples to room temperature in a desiccator and weigh to the nearest 0.01 mg. Record measurements on QAU form 3560.

11.0 INTERFERENCES

1) Characteristics of a sediment affecting survival, independent of chemical concentration.
2) Changes in chemical bioavailability as function of sediment manipulation or storage.
3) Presence of indigenous organisms.

12.0 QUALITY CONTROL CHECKS AND ACCEPTANCE CRITERIA

Minimum 90% survival of organisms in control group and measurable growth (relative to dry-weight of archive group) of organisms exposed to the control.

Conduct 96h, water-only reference toxicant test (cadmium chloride), with each lot of organisms.

Calibrate all measuring equipment used (thermometers, balances, meters) per established procedures.

13.0 CORRECTIVE ACTIONS AND CONTINGENCIES FOR OUT OF CONTROL DATA

Repeat any test which does not meet acceptance criteria. An individual test may be conditionally acceptable if specified conditions fall outside specifications, depending on degree of departure and test objectives. The acceptability of a test will depend on the professional judgement of the project director and regulatory authority. Tests deemed unacceptable must be re-run.

14.0 POLLUTION PREVENTION, WASTE MANAGEMENT AND SAMPLE DISPOSAL

Unless otherwise directed by project sponsor, place all remaining test material (used and unused) in sealed HDPE buckets and discard in waste collection container.

15.0 DOCUMENTATION

Document water quality parameters and survival counts in test notebook. Archive original data at PBS&J Environmental Toxicology Laboratory.

Record data on QAU 3560, QAU 6109, and QAU 7420a.

TABLE 9.1 SUMMARY OF TEST CONDITIONS FOR 28-D SEDIMENT TOXICITY TEST WITH N. arenaceodentata	
TEST TYPE:	whole sediment toxicity test; static-renewal
TEMPERATURE:	20 ± 1°C
SALINITY:	25 -32 ppt
LIGHT QUALITY:	Wide-spectrum florescent lights
ILLUMINANCE:	50-100 ft-c
PHOTOPERIOD:	16L:8D
TEST CHAMBER:	1L glass beaker or jar
SEDIMENT VOLUME:	~175 mL (2cm)
OVERLYING WATER VOLUME:	775 mL
RENEWAL OF OVERLYING WATER:	24h intervals, beginning on Day 0 (minimum), or as specified
SIZE AND LIFE STAGE OF POLYCHAETES:	2-3 week old juveniles
NUMBER OF ORGANISMS PER CHAMBER:	5
NUMBER OF REPLICATES PER TREATMENT:	5
FEEDING:	feed on days 0 through 28; 1mL TetraMarin®/Alfalfa suspension per test vessel
AERATION:	moderate, overnight before start of test and throughout duration of test; maintain ≥60% saturation of dissolved oxygen concentration
OVERLYING WATER:	clean seawater, natural or reconstituted water
OVERLYING WATER QUALITY:	Temperature, pH, ammonia, salinity, and DO of overlying water daily. Salinity, ammonia and pH of pore water
TEST DURATION:	28d
ENDPOINTS:	Survival and growth
TEST ACCEPTABILITY:	Minimal mean control survival of 90%

SOP No. 4020	Rev. No.: 2.4	Date: September 28, 2009
***Mysidopsis bahia* Chronic WET**		**Reference Method: EPA 1007.0**

1.0 PURPOSE AND APPLICABILITY

This procedure is used to estimate the chronic toxicity of effluents and receiving waters to the mysid shrimp, *Mysidopsis bahia*. The two endpoints measured in a chronic *M. bahia* test are survival and growth (weight).

2.0 REFERENCES

References listed in this section are incorporated into this SOP.

US EPA. *Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms*, 3rd edition. EPA-821-R-02-014, Test Method 1007.0.

SOP 5006: Calibration, Operation and Maintenance of the Orion Model 210A pH Meter

SOP 5007: Calibration, Operation and Maintenance of the Orion Model 410A pH Meter

SOP 5008: Calibration, Operation, and Maintenance of the Orion 3 Star pH Meter

SOP 5002: Calibration, Operation and Maintenance of the YSI Model 55 Dissolved Oxygen and Temperature System

SOP 5003; Calibration, Operation and Maintenance of the YSI Model 30 Handheld Salinity, Conductivity and Temperature System

3.0 DEFINITIONS

dead - Test organisms are “dead” if they exhibit (1) no movement and (2) no reaction to gentle prodding.

scheduled terminal time - time for test termination, calculated by adding test duration (measured in hours) to recorded test initiation time

critical dilution - concentration of effluent used in dilution series of toxicity test; effluent concentration representative of proportion of effluent in receiving water during critical low flow or critical mixing conditions.

4.0 HEALTH AND SAFETY CONSIDERATIONS

Effluents submitted for toxicity testing are potentially hazardous -- handle with appropriate care. Use standard laboratory personal safety equipment when handling effluents and receiving waters; at minimum, wear gloves at all times.

5.0 QUALITY ASSURANCE AND PLANNING CONSIDERATIONS

Variations from this procedure are not anticipated or encouraged. Justify study-specific amendments in study protocol, work plan, or test notebook, and evaluate (in writing) with respect to potential effects on this procedure.

6.0 RESPONSIBILITIES

Saltwater Testing Manager **AND** Laboratory Coordinator assure that assigned personnel are fully trained to perform this procedure.

Laboratory Technicians follow this procedure as specified.

7.0 TRAINING/QUALIFICATIONS

No specific training or qualifications, other than documented training to requirements of this SOP, are required; training records are current.

8.0 REQUIRED MATERIALS

- test samples (organisms must be exposed to at least three samples)
- receiving water and/or laboratory-prepared synthetic sea water
- pH meter, calibrate and use according to SOP #5005
- dissolved oxygen meter, calibrate and use according to SOP #5002
- salinity meter, calibrate and use according to SOP #5003
- thermometer, calibrate and use according to SOP #5012
- 2-L graduated cylinder
- 12oz disposable plastic cups
- computer generated random number list
- 5X7 cm Nitex® mesh loading net (400-500μm)
- test organisms (*Mysidopsis bahia,* 7d)
- newly-hatched *Artemia nauplii* in suspension
- large glass bowl
- 25X25 cm Nitex® mesh net (400-500μm)
- small metal forceps
- dissecting probe
- drying oven
- pre-weighed 1X1cm aluminum pans

9.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Store all effluents and receiving waters in darkened cooler at 0-6°C until use. Make sure that head space above sample is minimal. Time from sample collection to first use must not exceed 36 hours. Holding time for samples used in test renewals must not exceed 72h from sample collection**.** There may be holding time exceptions based on communication with the permitting authority.

10.0 METHOD

10.1 Test Samples

10.1.1 Do chemical analysis (pH, DO, salinity, conductivity, temperature, hardness, alkalinity, total residual chlorine and total ammonia) on aliquot of all samples used in toxicity testing. At minimum, measure total residual chlorine before sample is used in toxicity testing. De-chlorinate sample if specified in permit.

10.1.2 If samples are warmed to bring them to prescribed test temperature, supersaturation of DO may become a problem. If DO is greater than 100% saturation or lower than 4.0mg/L, aerate sample moderately until DO is within prescribed range. Once test is under way, aerate test solutions if the dissolved oxygen is < 4.0mg/L.

10.2 Dilution Water and Control Medium

Type of dilution water (laboratory-prepared seawater, generally 25ppt **OR** receiving water collected upstream and outside the influence of the outfall and salted to the appropriate salinity*****) used in effluent toxicity tests depends largely on test objective. Tests run with lab water as diluent include 100% lab water control; tests using receiving water include 100% lab water control **AND** 100% receiving water control.

***** In general, receiving water is not to be used if received at greater than 30 ppt. Must check with project manager or lab coordinator before using. Also, chlorine and salinity measurements must always be performed on receiving water samples before use.

10.3 Experiment Design

Mysid chronic tests are 7-day static renewal. Renew test solution daily. Use at least three effluent samples throughout test duration.

Conduct tests with five effluent concentrations (specified by permit) and one or more controls, as described above. Use 10 replicates (minimum: 8) for each test concentration and control.

10.4 Test vessels

Test vessels are 12oz disposable plastic cups.

10.5 Test organisms

Use mysids that are 7 days old at start of test -- 5 organisms per replicate.

10.6 Feeding

Feed 2 X day -- once before and once after solution renewal -- new-hatched *Artemia nauplii,* ~375 to each test cup.

10.7 Test Initiation

1) On Day 0, obtain organisms and verify that animals have acclimated to correct test temperature.
2) If necessary, warm sample in hot water bath to 26±1°.
3) Use HMM as needed to bring sample to prescribed salinity (see permit or scope of work).
4) Prepare 2.5L (250mL per test vessel) of each test concentration, according to permit specifications. Distribute solutions among test vessels.
5) Measure and record pH, DO, temperature and salinity.
6) Load 5 organisms into each test cup, using Nitex® mesh loading net.
7) Have second technician confirm that correct number of organisms are loaded, place cups on test bench according to random number.
8) Maintain test at 26±1°C with 16L:8D photoperiod at illuminance ~50-100fc.

10.8 Test Maintenance

1) Measure and record pH, DO, and temperature of old solution in one test chamber at each test concentration and in the control.
2) Do test renewals on Days 1-6.
3) Prepare 2L of each test concentration according to permit specifications and measure and record pH, DO, temperature and salinity.
4) Working with one treatment group at a time, pour out ~80% of test solution from each test cup into large glass bowl.
5) Count and record the number of surviving (not “dead”) organisms every 24h. Remove dead animals (see section 3.0 for “dead” criteria) and discard appropriately.
6) Clean test cups with plastic pipette to remove excess food, metabolic wastes or particulate matter that settles from effluent.
7) Re-fill test cups with newly-mixed solutions, and return to bench.

10.9 Test Termination

1) Terminate tests after 7d at scheduled terminal time ±2h, provided the test has been exposed to three samples.
2) Measure and record pH, DO, and temperature of test solutions.
3) Working with one treatment at a time, count and record number of surviving organisms from each test cup.
4) Working with one cup at a time, pour contents of each test cup onto 500µm mesh screen (approximately 25X25cm).
5) Rinse larvae with de-ionized water to wash away salts that might contribute to dry weight.
6) Using small forceps and dissecting probe, place surviving organisms on 1X1cm pre-weighed aluminum pan.
7) Place pans in drying oven overnight at 105°C.
8) On Day 8, remove pans from oven. Weigh and record weight of each pan on data form.

11.0 INTERFERENCES

Section not applicable.

12.0 QUALITY CONTROL CHECKS AND ACCEPTANCE CRITERIA

Acceptance criteria for control group:

- survival ≥80%
- average dry weight per surviving organism in control group ≥0.20 mg
- coefficient of variation between control replicates, **AND** between critical dilution replicates, ≤40% for both survival and growth
- reference toxicant LC50's within control limits (± 2sd from mean)

13.0 CORRECTIVE ACTIONS AND CONTINGENCIES FOR OUT OF CONTROL DATA

Rerun any tests that do not meet acceptance criteria.

14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

At test termination, dispose of test water in the sink; flush sink thoroughly with running tap water. Dispose of test cups in waste receptacle.

15.0 DOCUMENTATION

Document water quality parameters, survival counts, and test organism weights on QAU form #1600.

DRAFT

SOP No. 4042	Rev. No.: 1.0	Date: June 14, 2010

Earthworm (*Eisenia fetida*) Survival and Growth Soil Toxicity Test

1.0 PURPOSE, SCOPE AND APPLICABILITY

The purpose of this procedure is to establish a uniform method for conducting soil toxicity tests with the earthworm, *Eisenia fetida*, at the PBS&J laboratory. This procedure includes survival and growth endpoints for evaluating the toxicity of soils. This procedure is applicable to and may be used for projects where the objective is to evaluate soil toxicity; this procedure is not applicable for evaluating the potential for soil-borne contaminants to bioaccumulate.

2.0 REFERENCES

ASTM E 1676-04. Standard Guide for Conducting Laboratory Soil Toxicity or Bioaccumulation Tests with the Lumbricid Earthworm *Eisenia fetida* and the Enchytraeid Potworm *Enchytraeus albidus*. ASTM International. West Conshohocken, PA. April 2004.

3.0 DEFINITIONS

artificial soil—a synthetic soil, prepared with a specific formulation, designed to simulate a natural soil. Artificial soil may be used as a diluent medium to prepare concentrations of site or reference soil and may be used as a negative control medium.

clitellum—the fleshy "ring" or "saddle" of glandular tissue found on certain mid-body segments of oligochaete (Lumbricidae and Enchytraeidae) worms. It is the most visible feature of an adult earthworm or potworm and secretes the cocoon into which eggs and sperm are deposited.

diluent soil—the artificial or reference soil used to dilute site soils.

hydration water—water used to hydrate test soils to create an environment with a moisture level suitable for the species being tested. The water used for hydration is often test water; however, depending on the nature of the test being implemented, site surface water or groundwater may also be utilized for hydration.

negative control soil—artificial or reference soil to be used for evaluating the acceptability of a test.

reference soil—a field-collected soil that has physicochemical and biological properties as similar as possible to the site soil but does not contain the potentially toxic compounds of the site soil. It is used to describe matrix effects on the test in question. It may be used as a diluent medium to prepare concentrations of site soil and may be used as a negative control medium.

site soil—a soil collected from the field to be evaluated for potential toxicity. A site soil may be a naturally occurring soil or one that has been influenced by xenobiotics.

test soil—a soil prepared to receive a test organism. Site or reference soil mixed with artificial soil or reference soil mixed with site soil in known concentrations for evaluation are test soils. Artificial, site, or reference soils spiked with test materials such as chemicals, oils, or manufacturing products are test soils. Once a site, reference, or artificial soil is hydrated, even though it is not mixed with artificial or reference soil or spiked with a material, it may be called a test soil.

test water—water used to prepare stock solutions, rinse test organisms, rinse glassware, and apparatus or for any other purpose associated with the test procedures or culture of the test organism. Test water must be deionized or distilled water or better, such as reagent-grade water produced by a system of reverse osmosis, carbon, and ion-exchange cartridges.

4.0 HEALTH AND SAFETY CONSIDERATIONS

Field-collected soils may be contaminated with hazardous chemicals. Use of appropriate personal protection equipment (PPE), including gloves, aprons (or lab costs), dust masks (and in some cases, respirators) is recommended and may be required.

Specific information regarding potential hazards and risks may be available in a sponsor-prepared health and safety plan.

5.0 QUALITY ASSURANCE AND PLANNING CONSIDERATIONS

A sponsor-prepared Quality Assurance Project Plan (QAPP) may be available and should be consulted for details pertaining to laboratory tasks and activities.

Guidance provided in this procedure may be adapted to meet project-specific requirements. Adaptations of this procedure must be written in sufficient detail so as to establish th degree of variance and must be pre-approved by the laboratory's Technical Directory or Laboratory Director and project sponsor priot to implementation.

6.0 RESPONSIBILITIES

Special Projects Director-- will communicate specific project requirements, including quality assurance and health and safety concerns, to the laboratory staff.

Special Projects Supervisor–will schedule work and oversee staff assigned to work performance.

Technical Staff–will perform work in accordance with this procedure.

7.0 TRAINING/QUALIFICATIONS

No specific training or qualifications, other than documented training to the requirements of this SOP, are required. General training records for technical personnel assigned to tasks included in this procedure must be current.

8.0 REQUIRED MATERIALS

- 1 pt (463 cc) canning jars, with screw-rings and punched lids (1-2 mm hole)
- 2-L polyethylene mixing bowl
- polyethylene or teflon spatula or mixing spoon
- wash bottle
- forceps, teasing needles
- 250 mL glass beaker or disposable plastic cup
- 500 ml erlenmeyer flask
- 100 mm (mouth diameter) glass funnel
- 18.5 cm (diam) coarse filter paper (VWR No. 417)
- top-loading balance
- analytical balance
- 70 mm aluminum weigh pans (VWR No. 25433-085), 1 per test unit

9.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

Sample collection is typically performed by the project-sponsor or a third-party contractor and is covered by the project work plan (QAPP).

Samples received at the PBS&J laboratory are stored, in the original containers, in a darkened walk-in cooler maintained at 1-6°C.

10.0 METHOD

10.1 Experimental Design–Decisions concerning the various aspects of experimental design, such as the number of treatments and number of test containers and test organisms per container, will be based on the purpose of the test and the type of procedure that is to be used to calculate results. At a minimum, the earthworm soil toxicity test must include 5 replicates for test, reference or control sediment. At a minimum, each replicate treatment must include 10 fully-clitellate adult worms.

10.2 Test Organisms–*Eisenia fetida* (Savigny, 1826), Oligochaeta, is used for this procedure. Specimens may be obtained from cultures maintained within the testing facility or from an approved commercial vendor. The taxonomic status of each lot of organisms used in a test must be confirmed.

10.3 Test Conditions–Earthworm soil toxicity tests are conducted at 20±1°C under continuous light (~100 fc) provided by cool-white fluorescent light fixtures.

10.4 Test Containers–Glass containers (eg., 1 pt. canning jars) are used as testing containers for earthworm toxicity tests. Each test container is covered with a pierced (1- to 2-mm hole, to allow gas exchange) jar lid held in place with a screw ring.

10.5 Procedure

10.5.1 Determine Soil Moisture & Water-Holding Capacity

Moisture Content--Place clean 8 oz wide-mouth jar on top-loading balance and set tare weight to zero; add 100±0.5g well-mixed site, reference or artificial soil to the jar. Remove jar and re-set the balance to zero. Place the jar (with soil) back on the balance; measure and record the total weight of the jar and soil. Dry the sample for 24 hours at 100±5°C; cool in a dessicator and re-weigh the jar and sample. Cap the jar tightly to minimize moisture uptake if the sample will not be used immediately for further processing. Calculate the moisture content (MC) of the sample as,

$$MC(ml \cdot 100g^{-1}) \equiv IWW(g) - FDW(g)$$

where, IWW is the initial wet weight of the sample plus jar and FDW is the final dry weight of the sample plus jar.

Water-holding Capacity–Place 100 g of the dry sample into a 250 ml beaker. Add 100 mL deionized water and stir with a glass stir rod to ensure all sample particles are wetted and that slurry of soil and water exists. Fit a folded, coarse paper filter (VWR No. 417, 18.5 cm diam.) into a 100 mm glass funnel and hydrate the entire surface of the filter with 9-10 mL deionized water. Allow any excess water to drain away and measure the weight (nearest 0.01 g) of the funnel and hydrated filter paper. Place funnel in a 500-mL erlenmeyer flask and slowly pour the soil-water slurry onto the filter; rinse any soil remaining in the beaker and on the stir rod into the funnel with a minimal volume of deionized water. Cover the funnel tightly with aluminum foil and allow it to drain for 3 h at room temperature. Weigh the funnel, filter paper and soil to obtain the final weight. Determine the water-holding capacity (WHC) of the soil as,

$$WHC(ml \cdot 100g^{-1}) \equiv IW(g) - FW(g)$$

where, IW is the initial weight of the funnel and hydrated filter and dry soil, and FW is the final weight of the funnel, hydrated filter and wetted-and-drained soil.

10.5.2 Soil Hydration

Adjust the water content of each soil sample to 75% of its water-holding capacity with deionized water. Determine the amount (mL·100g^{-1}) of hydrating water (HW) required as,

$$HW \equiv (0.75xWHC_{ts}) - MC_{ts}$$

where THW_{ts} is the desired test soil hydration water and MC_{ts} is the existing test soil moisture content.

Place 2-L polyethylene mixing bowl on top-loading balance and set the balance to zero add 1000g test soil to the bowl. Add 10xHW volumes of deionized water to the bowl and mix thoroughly to uniformly wet the test soil. Re-weigh the bowl and hydrated soil and determine the total weight of the hydrated soil.

10.5.3 Pre-Test Set-up

The day before the toxicity test is started (Day -1), place test jars on a top-loading balance and set to zero; divide the hydrated test soils (~ 1/5th portions) evenly among five test jars. If large interstitial spaces of air occur in the soil matrix, remove the voids by pressing the soil with a suitable utensil, for example, a spatula, while trying not to compact the soil. Fix lids (with holes) on the jars and position the jars in the testing area; let stand over-night to achieve thermal equilibrium.

The day before the test is started (Day -1), remove sections of bedding material from the earthworm culture trays to a clean, sorting dish (eg., pyrex baking dish). Pick through the bedding material, selecting fully-clitellate adult specimens (Fig.1) of uniform length (largest specimen should be no more than about 10-20% longer than the shortest)

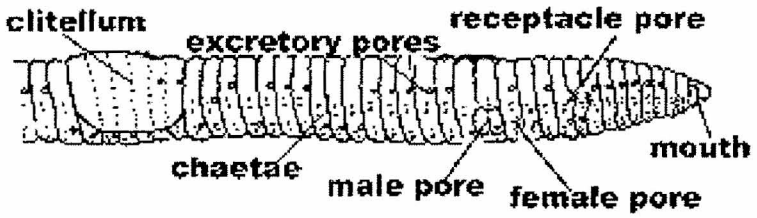

Fig. 1 Earthworm body structure, showing relationship of clitellum to anterior segments and reproductive pores[1].

[1] Conrad, Jim. Page title: *Earthworms.* Retrieved from **The Backyard Nature Website** at http://www.backyardnature.net/earthwrm.htm

Place each worm into a petri dish and rinse bedding material from external surfaces using a gentle stream of deionized water. Carefully lift each worm from the dish and place in groups of 10 worms onto a piece of wet filter-paper in a second petri dish. Cover the worms with a second, wetted filter disc; place the lid on the petri dish and let stand overnight to purge gut contents.

10.5.4 Test Initiation

On the day that the test is to be started, remove worms from the petri dishes, rinse to remove castings and blot dry on absorbent paper towel. Weigh each group of ten organisms; record weights on QAU Form nnn.

Test organisms are placed into the test containers after the overnight equilibration; this constitutes the beginning of the test (Day 0). The test organisms are placed on the surface of the soil and allowed to burrow because a lack of burrowing is considered a response possibly due to the presence of toxic compounds.

10.5.6 Test Duration

The test begins when test organisms are first placed in the test containers and continues for 14 or 28 days as specified in the project workplan.

10.5.7 Test Measurements

Temperature should be monitored for the duration of the test. A continuous temperature recorder (or a continuous temperature/humidity recorder) with a seven-day chart can be placed in the test chamber and changed as necessary.

pH should be measured at the beginning of the test in subsamples taken from the batch preparations and at the end of the test in subsamples from replicates of the various concentrations (or test groups). Care should be exercised to avoid a sample of soil containing dead worms.

Percent moisture may be measured at the beginning and end of the test from subsamples.

10.5.8 Food

Earthworms obtain nutrients from the surface of ingested soil particles; therefore, supplemental food is not generally required for tests of up to 28 days duration.

10.5.9 Toxicity Endpoint Assessment

At the end of the test (i.e., after 14 or 28 days continuous exposure), test soil and organisms are emptied onto a flat surface, and live organisms are removed and counted. Mortality is defined as a lack of response to a gentle mechanical stimulus, for example, touch with a small spatula or glass rod, to the anterior end of the worm. Earthworms may die and decompose within a 14-day testing period, so if all of the individuals are not accounted for at the end of the test, it may be assumed that they died and decomposed completely. Record the number of surviving worms on QAU Form nnn.

If biomass is to be evaluated, rinse the surviving earthworms and place onto moist filter paper (as described in 10.5.3) for gut purging. After 24h, rinse the worms carefully to remove castings, place onto a square of nylon bolting cloth and immerse in iced-water for about 20 seconds to quick-kill the worms, then blot dry on paper towels. Place the worms onto pre-weighed aluminum pans and determine total (group) weights. Record pan tare weights and total weights on QAU Form nnn.

Compare survival and biomass metrics for worms exposed to test, reference and negative control soils using appropriate statistical methods.

11.0 INTERFERENCES

Limitations to the methods described in this procedure might arise and influence soil toxicity test results and complicate data interpretation. The following factors should be considered when testing soils:

- The alteration of field samples in preparation for laboratory testing (for example, transport, screening, or mixing).
- Interaction among chemicals present in the soil.
- Addition of food to test containers may affect the results of a toxicity test, but it may be necessary to feed the test organisms in long-duration tests.
- The natural geochemical properties of test soil collected from the field might not be within the tolerance limits of the test species.
- Field-collected soils may contain indigenous organisms including (*1*) the same or closely related species to that being tested and (*2*) microorganisms (for example, bacteria and molds) and algae species that might grow in or on the soil and test container surfaces.

12.0 QUALITY CONTROL CHECKS AND ACCEPTANCE CRITERIA

An earthworm toxicity test is invalid if mean survival of organisms exposed to the negative control soil is less than 90%.

13.0 CORRECTIVE ACTIONS AND CONTINGENCIES FOR OUT OF CONTROL DATA

Re-run any toxicity test which does not meet minimum acceptance criteria for control survival.

14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Unless otherwise directed by project sponsor, place remaining test material (used and unused) in sealed HDPE buckets and discard in waste collection container.

15.0 DOCUMENTATION

Document test condition (temperature, pH,moisture, etc.), survival counts, tare weight, and total weight measurements on QAU Form nnn. Document changes to protocol in test notebook. Archive original data at PBS&J Environmental Toxicology Laboratory.

DRAFT

SOP	Earthworm (*Eisenia fetida*) Survival and Growth Soil Toxicity Test	SOP No.	4042
		Date	6/16/10

Origination and Acceptance:

	Name	Signature	Date
Originator:	James D. Horne		
Quality Assurance Unit:	Susan Gregory		
Laboratory Director:	Faust R. Parker, Jr.		

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.

Client/Project Name:		Login #:		Job #:	

Eisenia fetida 28d Test Condition Summary - ASTM E 1676-04; PBS&J SOP No. 4042			
Test Material:	Soil	Test Type:	28d Survival & Growth
Temperature:	20±1°C	Photoperiod:	24L:0D (~100 fc)
Test Chamber:	1 pt. jar w/ punched-lid & ring	No. Replicates:	5
Test Organism:	***Eisenia fetida***	Organism Source:	
Date Received:		Organism Batch No.:	
Age Class:	adults (300-600 mg·worm^{-1}; largest specimen no more than 10-20% longer than shortest)	No. Organisms/Rep:	10
Control Soil:	Artificial soil; 10% sphagnum peat moss (screened @ 2000 µm), 20% kaolin clay, 70% silica sand (#70 grade)		
Feeding Schedule:	Project specific	Food Type:	Alfalfa flour

Initiation Date:		Termination Date:	
Initiation Time:		Termination Time:	
Initials:	/	Initials:	/

Comments:

Final Review Completed By:_____

Initial Review:_____/_____

Data correction codes: IE-incorrect entry; WC- wrong column; WR-wrong row; TN-transposed number; ONV-organism not visible; SC-spilled cup; PB-pathogenic bacteria

Client/Project Name:		Login #:		Job #:	

MOISTURE CONTENT DETERMINATION

Sample ID	Initial Wet Weight (IWW)	Final Dry Weight (FDW)	Moisture Content (MC) MC(ml·100g^{-1})10 = IWW(g) - FDW(g)
Date Drying Initiated:		Date Drying Terminated:	
Time Drying Initiated"		Time Drying Terminated:	
Oven Temp °C (Actual/Off-set):		Oven Temp °C (Actual/Off-set):	
Initials:		Initials:	

WATER-HOLDING CAPACITY DETERMINATION

Sample ID	Initial Weight (IW)	Final Weight (FW)	Water Holding Capacity (WHC) WHC(ml·100g^{-1})10 = IWW(g) - FDW(g)	Volume Hydration Water Added (mL·kg^{-1})
Initials:		Date/Time	/	

Initial Review:_____/_____

Data correction codes: IE-incorrect entry; WC- wrong column; WR-wrong row; TN-transposed number; ONV-organism not visible; SC-spilled cup; PB-pathogenic bacteria

Client/Project Name:		Login #:		Job #:	

Eisenia fetida **- Survival**

Treatment	Rep	Day 0	Day 28		Treatment	Rep	Day 0	Day28	
			1st Count	2nd Count				1st Count	2nd Count
	A					A			
	B					B			
	C					C			
	D					D			
	E					E			
	A					A			
	B					B			
	C					C			
	D					D			
	E					E			
	A					A			
	B					B			
	C					C			
	D					D			
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	B					B			
	C					C			
	D					D			
	E					E			
	A					A			
	B					B			
	C					C			
	D					D			
	E					E			
Tech Initials					Tech Initials:				

Initial Review: ______/______

Data correction codes: IE-incorrect entry; WC- wrong column; WR-wrong row; TN-transposed number; ONV-organism not visible; SC-spilled cup; PB-pathogenic bacteria

Client/Project Name:		Login #:		Job #:	

WET TISSUE WEIGHT - *Eisenia fetida*

SAMP ID	REP	PAN NO.	TARE WT (g)	TOTAL WT (g)	SAMP ID	REP	PAN NO.	TARE WT (g)	TOTAL WT (g)
	A	1				A	26		
	B	2				B	27		
	C	3				C	28		
	D	4				D	29		
	E	5				E	30		
	A	6				A	31		
	B	7				B	32		
	C	8				C	33		
	D	9				D	34		
	E	10				E	35		
	A	11				A	36		
	B	12				B	37		
	C	13				C	38		
	D	14				D	39		
	E	15				E	40		
	A	16				A	41		
	B	17				B	42		
	C	18				C	42		
	D	19				D	44		
	E	20				E	45		
	A	21				A	46		
	B	22				B	47		
	C	23				C	48		
	D	24				D	49		
	E	25				E	50		
QA/QC (pans)					Comments:				

Initial Review: _____/_____

Data correction codes: IE-incorrect entry; WC- wrong column; WR-wrong row; TN-transposed number; ONV-organism not visible; SC-spilled cup; PB-pathogenic bacteria

Appendix C

Quality Assurance Manuals

Appendix C-1

PBS&J - Quality Assurance Manual

Volume 1
QUALITY ASSURANCE POLICY

PBS&J
Environmental Toxicology Laboratory
888 West Sam Houston Parkway South, Suite 110
Houston, TX 77042-1917
Tel: 713-977-1500
Fax: 713-977-9233

Faust R. Parker, Jr., Ph.D.
Vice President & Division Manager
Laboratory Director

James D. Horne
Technical Director

Susan Bunch
Quality Assurance Manager

Rachael Brown
Laboratory Supervisor

Matt Matthews
Client Services Manager

DOCUMENT CONTROL NOTICE

Information contained in this document is the property of the PBS&J Environmental Toxicology Laboratory. This manual is not to be copied in any part or form or communicated for the use of any other party.

The PBS&J Quality Assurance Unit Manager maintains absolute responsibility and authority for the distribution, maintenance and re-call of this quality assurance manual.

Upon demand, or cessation of need on the part of the holder of record, this controlled copy must be returned to PBS&J Environmental Toxicology Laboratory.

CONTROLLED COPY NO.: _____

THIS MANUAL COPY IS RECORDED AS BEING ON LOAN TO:

NAME: _____

TITLE: _____

COMPANY: _____

ADDRESS: _____

DISTRIBUTION DATE: _____

GOAL

The PBS&J goal is to meet world class standards for the mutual benefit of our customers and employees and to be recognized nationally as the environmental toxicology service laboratory of choice.

MISSION

The mission of the PBS&J Environmental Toxicology Laboratory is to provide the highest-quality legally defensible data, exceptional client service, and the most comprehensive range of capabilities in the environmental toxicology testing industry.

OPERATING PHILOSOPHY

PBS&J is committed to a management system that makes quality a basic business principle. The strategy is based on customer satisfaction and is achieved through development of a clear understanding of internal and external customer requirements and, then, meeting the customer's needs on time.

Conformance to regulatory authority, as well as our customer's requirements and expectations, is the responsibility of all employees at PBS&J.

Quality assurance systems, procedures and practices are developed, reviewed and changed with participation of all employees in a continuous improvement effort.

MANAGEMENT POLICY STATEMENT

It is the policy of the PBS&J Environmental Toxicology Laboratory management to fully support and to provide the necessary resources for continual implementation of the quality assurance system.

Management at all levels will participate in quality assurance activities as incorporated into daily functional requirements.

No work product will be shipped to the customer until it's quality and conformance to customer specifications can be assured.

Management will assess the effectiveness of the quality system on a regular basis and direct internal efforts towards continual improvement.

The PBS&J management is committed to full compliance with the NELAC standards, to production of test data of known and documented quality, and to the quality assurance system outlined in this manual and supporting documents. Management will ensure this policy is communicated, understood, implemented and maintained at all levels within the organization.

Faust R. Parker, Jr., Ph.D.
Vice President & Division Manager
Director, PBS&J Environmental Toxicology Laboratory

10-10-08
Date

TABLE OF CONTENTS

PART I - GENERAL

1.0 FORWARD

1.1 PBS&J Environmental Toxicology Laboratory provides toxicity testing and consulting services (the work product) to support wastewater discharge permit requirements (eg., whole effluent toxicity tests and toxicity identification and reduction evaluations); marine and freshwater whole sediment toxicity tests and bioaccumulation assessments; and environmental fate and effects testing of industrial and consumer products, including drilling fluid systems and additives.

1.2 This Quality Assurance Manual describes the Quality System implemented at the PBS&J Environmental Toxicology Laboratory, with business operations at:

888 West Sam Houston Parkway South, Suite 110
Houston, Texas 77042-1917

1.3 The objective of the Quality System is to (1) prevent non-conformance through planning and project management, (2) provide for the prompt detection of non-conformance which may result in unsatisfactory quality, and (3) assure timely and effective Corrective Action.

1.4 This Quality System, designed and developed in conjunction with Managerial functions, establishes an effective and economical system for assuring work product quality. The Quality System embodies (1) Quality Assurance Policy [Vol. 1]; (2) Quality Assurance Procedures [Vol. 2]; (3) Standard Operating Procedures [Vol. 3]; and, (4) a system of records to document compliance to Quality System elements and conformance of the work product to specification.

1.5 It is PBS&J's Policy to provide full compliance with this Quality System throughout all phases of contract performance and to ensure that only acceptable work products are presented to the Customer.

2.0 QUALITY ASSURANCE UNIT

2.1 To ensure implementation and full compliance with the Quality System, PBS&J has established the Quality Assurance Unit (QAU).

2.2 The QAU consists of a part-time Manager. The QAU reports directly to the Laboratory Director and is responsible for the management of the Quality System.

2.3 The QAU monitors overall implementation of the Quality Assurance Program through performance and systems audits, and review of laboratory work products prior to distribution. The Quality Assurance Unit representatives are vested with the independence necessary to carry out their assigned responsibilities, including authorization from laboratory management to prevent delivery of nonconforming work until satisfactory corrective action has been taken.

2.4 PBS&J management has appointed Susan Bunch as the QAU Manager.

Faust R. Parker, Jr., Ph.D.
Laboratory Director

10-10-08
Date

3.0 CERTIFICATION

COMPANY CERTIFICATION PAGE

We hereby certify that this Quality Assurance Manual accurately and adequately describes the Quality System implemented at PBS&J for the provision of a Quality System to meet the laboratory accreditation requirements of the State of Texas Commission on Environmental Quality and the State of Louisiana Department of Environmental Quality. Certificates and scopes of accreditation are presented in Appendix A.

Susan Bunch
QAU Manager

10/10/08
Date

Faust R. Parker, Jr., Ph.D.
Laboratory Director

10-10-08
Date

4.0 QUALITY ASSURANCE MANUAL

Name of Firm: PBS&J

Division: Environmental Toxicology Laboratory

Address: 888 West Sam Houston Parkway South, Suite 110
Houston, TX 77042-1917

AMENDMENT CERTIFICATION

I hereby certify that this Manual has been reviewed and amended as necessary to reflect the current Quality System.

QAU Manager	Date	Laboratory Director	Date	Revision No.
Susan C Bunch	10/10/08		10-10-08	3.1

5.0 AMENDMENT PROCEDURE

5.1 The Quality Assurance Manual (QAM) shall be amended to reflect any changes to PBS&J's capability, location or Quality System.

5.2 The QAU Manager shall submit the QAM amendments to all persons holding controlled copies of the QAM, accompanied by a completed QAM Amendment Certification Page.

5.3 When a single amendment affects fifty percent (50%) or more of the QAM content, or when a maximum of ten (10) amendments are exceeded, the QAM shall be re-issued.

5.4 The QAU Manager is responsible for the maintenance of the QAM as described in this section and for reviewing the QAM annually.

5.5 Amendments to the QAM shall be recorded below.

REVISION/ AMENDMENT NO.	PAGES AFFECTED	DESCRIPTION	DATE
Rev 0	All	Initial Issue	12/23/1998
Rev 1	All	Major re-organization, with incorporation of CompQAP # 980176 and Florida Department of Environmental Protection Standard Operating Procedures (DEP-QA-001/92)	09/30/1999
Rev 2.0	All	Major Reorganization, with incorporation of Quality Assurance Procedures	03/26/2001
Rev 2.1	9, 10, 11, 17	CompQAP no longer applicable, laboratory certified by NELAC approved accrediting authority	07/01/2001
Rev 2.2	1, 7, 8, 9, 10,11,12,16	QAU Manager personnel change, revised organizational chart, addition of QAP17.	05/23/2003
Rev 2.3	1, 8, 9, 12	Laboratory supervisor personnel change, addition of LELAP certification, revised organizational chart	11/02/2005
Rev 3.0	All	Major re-organization; addition of TCEQ accreditation (primary authority)	08/31/2007
Rev 3.1	9, 21-29, 31	Revised Amendment Certification, current accreditation certificates inserted, revised organizational chart.	10/10/08

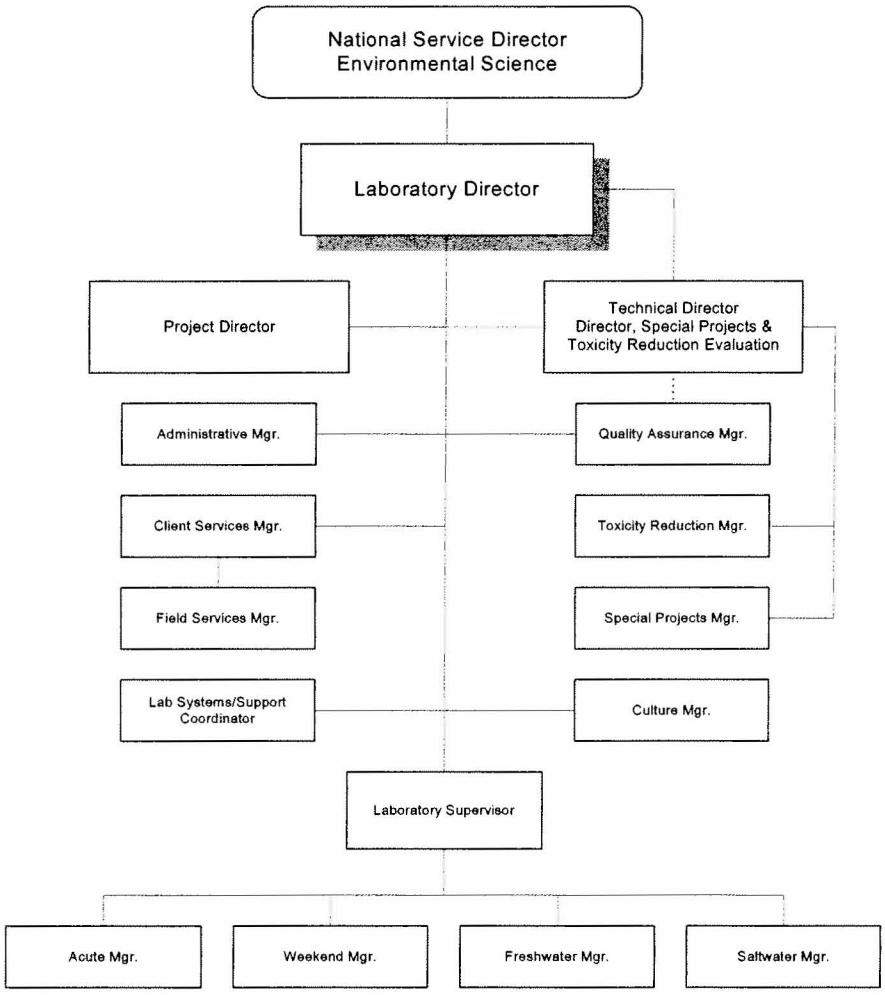
PART II – ORGANIZATION & FACILITIES

1.0 ORGANIZATION STRUCTURE

The PBS&J Environmental Toxicology Laboratory is a division of PBS&J Environmental Sciences and reports to Cecilia Green, Senior Vice President and National Service Director. The organization structure of the PBS&J Environmental Toxicology Laboratory is presented below.

Job descriptions for key laboratory personnel are provided in Appendix B.

PBS&J Environmental Toxicology



2.0 ORGANIZATION & AUTHORITY POLICY

2.1 Quality is the responsibility of all PBS&J employees and agents.

2.2 All personnel shall be accountable for the quality of work performed through their individual assignments and functional responsibilities.

2.3 Employees shall be responsible for reporting any non-conformance to the QAU Manager.

2.4 The QAU Manager shall maintain the organizational freedom and authority for:

(a) Full implementation of the Quality System.

(b) Identifying and recording Quality problems.

(c) Initiating, recommending or providing solutions through designated channels.

(d) Verifying implementation of solutions.

(e) Controlling further processing and delivery of non-conforming work products, until the condition has been corrected.

2.5 The QAU Manager shall address all problems which cannot be resolved with other members of PBS&J Management to the Laboratory Director for resolution.

2.6 The Laboratory Director is responsible for the review of the Quality System and for the verification of resources including trained personnel.

2.7 Management review and verification of the Quality System is conducted annually as a minimum.

3.0 QUALITY BOARD

3.1 PBS&J has organized a Quality Board (QB) to advise the Laboratory Director on matters pertaining to quality.

3.2 The QB is comprised of the following Managerial Functions:

(a) Technical Director

(b) Client Services Manager

(c) Laboratory Supervisor

(d) Quality Assurance Manager

3.3 Various PBS&J personnel will participate in QB activities as requested by the QB.

3.4 The QB determines areas for quality and productivity improvement and presents them to the Laboratory Director for consideration.

3.5 QB activities include:

(a) Review, analysis and disposition of non-conformance reports.

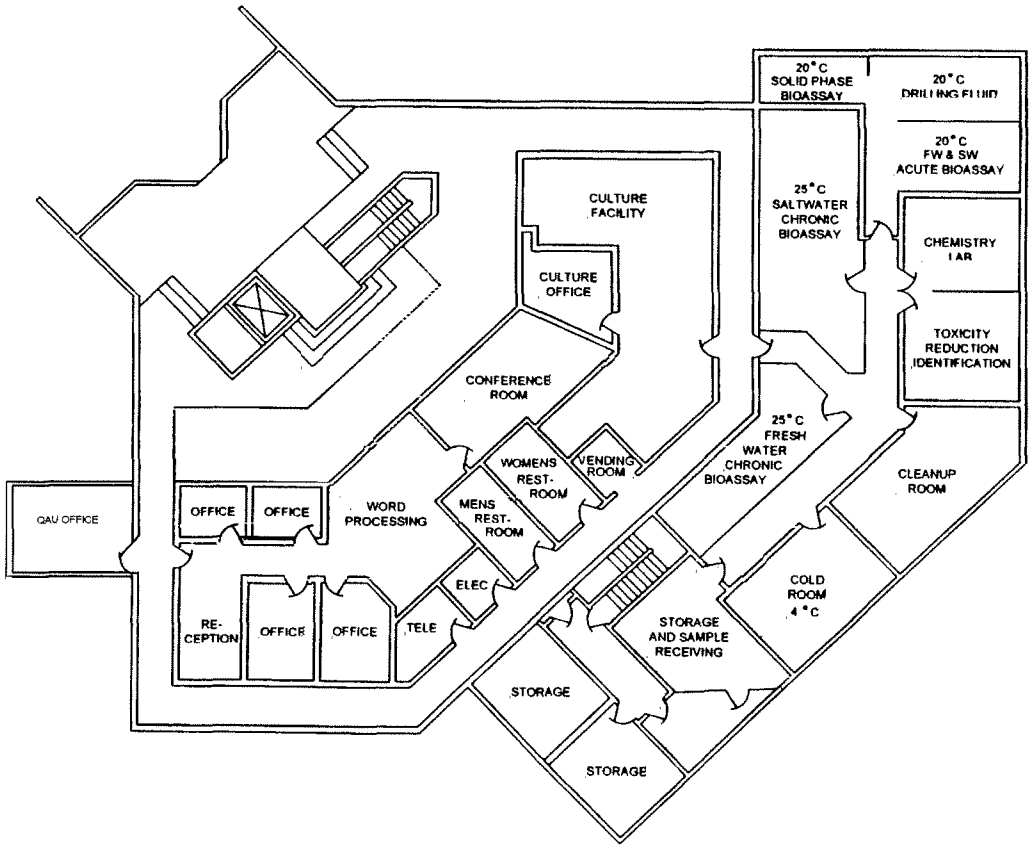
(b) Review of Corrective Actions.

(c) Review and analysis of Quality Records.

(d) Review and analysis of Audit results.

4.0 FACILITIES

PBS&J occupies approximately 9,000 square feet in a multi-tenant office building. The laboratory floorplan is depicted below.



PART III – QUALITY ASSURANCE PROCEDURES

Quality Assurance Procedures (QAPs) are provided in Volume 2. The QAPs are designed to be reviewed and/or revised independent of each other; therefore, pagination within Volume 2 of the Quality Assurance Manual is not sequential.

QAP #	Title
1	Organization and Management
2	Quality System - Establishment
3	The Quality Assurance Manual and Related Documents
4	Quality Systems Audits and Corrective Actions
5	Quality Systems - Essential Quality Control Procedures
6	Personnel
7	Physical Facilities - Accommodation and Environment
8	Equipment, Reference Materials, Measurement Traceability, and Calibration
9	Test Methods and Standard Operating Procedures
10	Sample Handling, Sample Acceptance and Sample Receipt
11	Records
12	Evidentiary Custody and Documentation
13	Laboratory Report Format and Contents
14	Subcontracting Analytical Services
15	Outside Support Services and Supplies
16	Complaints
17	Coordination of Quality Control Practices
18	Data Integrity

PART IV – STANDARD OPERATING PROCEDURES

Standard Operating Procedures (SOPs) are provided in Volume 3. The SOPs are designed to be reviewed and/or revised independent of each other; therefore, pagination within Volume 3 of the Quality Assurance Manual is not sequential.

SOP #	TITLE
1001	Reference Toxicant Program
1002	Preparation of SOPs
1003	Non-conformance
1004	Vendor Approval
1005	Managerial Review and the Quality Board
1006	Audits
1007	Demonstration of Capability
1008	Health & Safety Audits
1009	Lab Technician General Training
1010	Culturist Training
1011	Management of Change
2001	Sample Check in
2002	Receipt, storage and use of standards and reagents
2003	Collection of Intermediate Samples
2004	Sample Composting
3001	Chironomus tentans Food Preparation and Feeding
3010	Artemia nauplii - Preparation for Feed
3020	Selenastrum capricornutum Culture
3030	Isochrysis galbana (marine algae) preparation & feeding
3040	YCT Preparation
3050	Culture of Branchionus plicatilis (Rotifer) for Feed
3060	Daphnia species food preparation
3070	Flake food storage and use
3080	L plumulosus Food Preparation and Feeding
3090	Evaluation of New Food used in testing and culturing
3110	Pimephales promelas Culture Practices
3120	Daphnia magna Culture Practices
3130	Dapnia pulex Culture Practices
3140	Ceriodaphnia dubia Culture Practices
3210	Menidia beryllina Culture Practices

SOP #	TITLE
3231	Mysidopsis bahia production system culture practices
3232	Mysidopsis bahia Grow-out Systems Culture Practices
3233	Mysidopsis bahia post larval culture practices
3310	Nitrobacter and Nitrosomonas Factory Culture Practices
3320	Eheim Filters in Culture
3340	Seawater preparation and maintenance of mixing tank
3350	Taxonomic Identification
4001	Static Sheen Test
4002	SDF Ammended Sediments Preparation
4003	Leptocheirus plumulosus 10d Amended Sediment
4004	Pimephales promelas embryo larva study
4005	TIE Phase I
4006	28d Biodeg Seawater
4007	28d Closed Bottle
4008	Selanastrum capricornutum growth test (Method 1003.0)
4009	Marine algae growth inhibition test
4010	Preparation of a water accomodated fraction (WAF)
4012	PI Modified Chronic Pimephales promelas
4013	Hyalella azteca 10d Sediment Survival & Growth (ASTM E 1706)
4014	Chironomus tentans 10d Sediment Survival & Growth (ASTM E 1706)
4015	Daphnia pulex Acute WET (EPA 2021.0)
4016	Pimephales promelas Acute WET (EPA 2000.0)
4017	Mysidopsis bahia Acute WET (EPA 2007.0)
4018	Menidia beryllina Acute WET (EPA 2006.0)
4019	Cyprinodon variegatus Acute WET (EPA 2004.0)
4020	Mysidopsis bahia Chronic WET (EPA 1007.0)
4021	Pimephales promelas Chronic WET (EPA 1000.0)
4022	Ceriodaphnia dubia Chronic WET (EPA 1002.0)
4023	Menidia beryllina Chronic WET (EPA 1006.0)
4024	Cyprinodon variegatus Chronic WET (EPA 1004.0)
4025	O mykiss Acute WET (EPA 2019.0)
4026	Mysidopsis bahia 10d Sediment Survival
4027	Ampelisca abdita 10d USACE
4028	Paleomenetes pugio 10d Sed
4029	Ampelisca abdita 10d Sediment Survival (ASTM E 1367)

SOP #	TITLE
4030	Cyprinella leedsi Acute WET (EPA 2000.0)
4031	Ceriodaphnia dubia Acute WET (EPA 2002.0)
4032	Nereis virens 28d bioaccumulation (ASTM E 1688)
4033	Macoma nasuta 28d bioaccumulation (ASTM E 1688)
4034	Lumbriculus variegatus 28d bioaccumulation (EPA 100.3)
4035	Daphnia magna Chronic WET
4036	Mercenaria merceneria 28d bioaccumulation (ASTM E 1688)
4037	Preparation of SPP Using Dredged Material
4038	Reverse Phase Extraction (RPE) Test for Free Oil Contamination
4039	Mysidopsid bahia, Low Salinity Acclimation
4040	Ampelisca abdita and Americamysis bahia 7d
4041	Leptocheirus plumulosus 10d Sediment Survival (ASTM E 1367)
4042	Mercenaria mercenaria 7d WST
4043	Corbicula fluminea 28d bioaccumulation (ASTM E 1688)
4044	Hyalella azteca 10d Sediment Survival & Growth (EPA 100.1)
4045	Chironomus tentans 10d Sediment Survival & Growth (EPA 100.2)
4046	Elutriate Preparation for Water Column Bioassay
5001	Incident illuminance
5002	Measuring D.O. and calibration of meter
5003	Measuring SCT and calibration of SCT meter YSI model 30
5004	Measuring SCT and calibration of SCT meter YSI model 3100
5006	pH Meter Orion 210A
5007	pH Meter Orion 410A
5008	pH Meter Orion 3 Star
5009	Algae Density by Hemocytometer
5010	Algae Density by Spec
5012	Laboratory Thermometers Calibration & Use
5013	Ohaus Analytical balance; use and calibration
5014	Class S Weights Use and Maintenance
5015	Fluoride Measurement using Hach DR DR/3000 Spec.
5016	Ammonia probe
5017	Calibration & Operation of YSI 3256 Conductivity Cell
5020	Equipment Maintenance Scheduling
5021	Facility Maintenance-outside service
6001	Determination of total hardness

SOP #	TITLE
6002	Determination of total alkalinity
6003	Determination of total residual chlorine
6004	Sample Dechlorination
7001	Final data review and packaging
7002	Initial Data Review
7003	Electronic Document Storage
7004	Data Corrections
9001	Laboratory temperature control
9002	Acid bath system, use and maintenance
9003	Glass and plastic ware cleaning
9004	Laboratory photoperiod verification
9005	Synthetic seawater transfer and acceptability
9006	Nalgene Tank Maintenance
9007	Synthetic Freshwater Preparation
9008	Water Vessel and Eheim Maintenance
9009	Sample Storage and Disposition
9010	Verification of Reagent Grade Water Quality
9011	Sample Kit Preparation
9012	ISCO Model 3700 Portable Sampler

Appendix A - Accreditations

Texas Commission on Environmental Quality

NELAP-Recognized Laboratory Accreditation is hereby awarded to

PBS&J ENVIRONMENTAL TOXICOLOGY LABORATORY
888 WEST SAM HOUSTON PKWY. SUITE 110
HOUSTON, TX 77042-1917

in accordance with Texas Water Code Chapter 5, Subchapter R, Title 30 Texas Administrative Code Chapter 25, and the National Environmental Laboratory Accreditation Program.

The laboratory's scope of accreditation includes the fields of accreditation that accompany this certificate. Continued accreditation depends upon successful ongoing participation in the program. The Texas Commission on Environmental Quality urges customers to verify the laboratory's current accreditation status for particular methods and analyses.

Certificate Number: T104704202-08-TX
Effective Date: 7/1/2008
Expiration Date: 6/30/2009

Executive Director
Texas Commission on Environmental Quality

Texas Commission on Environmental Quality

NELAP - Recognized Laboratory Fields of Accreditation

PBS&J Environmental Toxicology Laboratory
888 West Sam Houston Pkwy.
Suite 110
Houston, TX 77042-1917

Certificate T104704202-08-TX
Issue Date: 7/1/2008
Expiration Date: 6/30/2009

These fields of accreditation supercede all previous fields. The Texas Commission on Environmental Quality urges customers to verify the laboratory's current accreditation status for particular methods and analyses.

Matrix: Solid and Chemical Materials

Category / Method: ASTM E1367-03

Analytes:	Code	AA	Analytes:	Code	AA
Toxicity	10338	TX			

Category / Method: ASTM E1688-00a

Analytes:	Code	AA	Analytes:	Code	AA
Bioaccumulation	10339	TX			

Category / Method: ASTM E1706-05

Analytes:	Code	AA	Analytes:	Code	AA
Toxicity	10338	TX			

Category / Method: EPA 600-R-99-064

Analytes:	Code	AA	Analytes:	Code	AA
Bioaccumulation	10339	TX	Toxicity	10338	TX

Category / Method: EPA 821-R-02-012

Analytes:	Code	AA	Analytes:	Code	AA
Acute toxicity	3300	TX			

Category / Method: EPA 821-R-02-013

Analytes:	Code	AA	Analytes:	Code	AA
Chronic toxicity	3325	TX			

Category / Method: EPA 821-R-02-014

Analytes:	Code	AA	Analytes:	Code	AA
Chronic toxicity	3325	TX			

Category / Method: EPA 823-B-98-004

Analytes:	Code	AA	Analytes:	Code	AA
Toxicity	10338	TX			

Matrix: Non-Potable Water

Category / Method: EPA 1000.0

Analytes:	Code	AA	Analytes:	Code	AA
Aquatic Toxicity, Chronic	10342	TX			

Category / Method: EPA 1002.0

Analytes:	Code	AA	Analytes:	Code	AA
Aquatic Toxicity, Chronic	10342	TX			

Category / Method: EPA 1003.0

Analytes:	Code	AA	Analytes:	Code	AA
Aquatic Toxicity, Chronic	10342	TX			

Texas Commission on Environmental Quality

NELAP - Recognized Laboratory Fields of Accreditation

PBS&J Environmental Toxicology Laboratory
888 West Sam Houston Pkwy.
Suite 110
Houston, TX 77042-1917

Certificate T104704202-08-TX
Issue Date: 7/1/2008
Expiration Date: 6/30/2009

These fields of accreditation supercede all previous fields. The Texas Commission on Environmental Quality urges customers to verify the laboratory's current accreditation status for particular methods and analyses.

Matrix: Non-Potable Water

Category / Method: EPA 1004.0

Analytes:	Code	AA	Analytes:	Code	AA
Aquatic Toxicity, Chronic	10342	TX			

Category / Method: EPA 1006.0

Analytes:	Code	AA	Analytes:	Code	AA
Aquatic Toxicity, Chronic	10342	TX			

Category / Method: EPA 1007.0

Analytes:	Code	AA	Analytes:	Code	AA
Aquatic Toxicity, Chronic	10342	TX			

Category / Method: EPA 2000.0

Analytes:	Code	AA	Analytes:	Code	AA
Aquatic Toxicity, Acute	10341	TX			

Category / Method: EPA 2002.0

Analytes:	Code	AA	Analytes:	Code	AA
Aquatic Toxicity, Acute	10341	TX			

Category / Method: EPA 2004.0

Analytes:	Code	AA	Analytes:	Code	AA
Aquatic Toxicity, Acute	10341	TX			

Category / Method: EPA 2006.0

Analytes:	Code	AA	Analytes:	Code	AA
Aquatic Toxicity, Acute	10341	TX			

Category / Method: EPA 2007.0

Analytes:	Code	AA	Analytes:	Code	AA
Aquatic Toxicity, Acute	10341	TX			

Category / Method: EPA 2021.0

Analytes:	Code	AA	Analytes:	Code	AA
Aquatic Toxicity, Acute	10341	TX			

NELAP RECOGNIZED

STATE OF LOUISIANA
DEPARTMENT OF ENVIRONMENTAL QUALITY

Is hereby granting a Louisiana Environmental Laboratory Accreditation to

DEQ LOUISIANA

PBS&J Environmental Toxicology Laboratory
888 West Sam Houston Pkwy, Suite 110
Houston, TX 77042-1917

THE NELAC INSTITUTE

Agency Interest No. 115286

According to the Louisiana Administrative Code, Title 33, Part I, Subpart 3, LABORATORY ACCREDITATION, the State of Louisiana formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed in the attachment.

The laboratory agrees to perform all analyses listed on this scope of accreditation according to the NELAC standards and Part I, Subpart 3 requirements and acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part I. Please contact the Department of Environmental Quality, Louisiana Environmental Laboratory Accreditation Program (LELAP) to verify the laboratory's scope of accreditation and accreditation status. Accreditation by the State of Louisiana is not an endorsement or a guarantee of validity of the data generated by the laboratory.

To be accredited initially and maintain accreditation, the laboratory agrees to participate in two single-blind, single-concentration PT studies, where available, per year for each field of testing for which it seeks accreditation or maintains accreditation as required in LAC 33:I.4711.

Melvin C. Mitchell Sr., Accreditation Officer
Louisiana Environmental Laboratory Accreditation Program

Certificate Number: 04087
Expiration Date: June 30, 2009
Issued On: July 1, 2008

Louisiana Environmental Laboratory
LELAP
Accreditation Program

Laboratory Scope of Accreditation

Organization

04087 (713) 977-1500
PBS&J Environmental Toxicology Laboratory
888 West Sam Houston Pkwy., Suite 110
Houston, TX 77042-1917

Solid and Chemical Materials Certification

Method Code	Method Ref	Analyte	Status	Date Effective	Type	AA
2237	EPA 823/B-98-004	Americamysis bahia	Accredited	7/29/2008	NELAP	TX
2319	ASTM E 1367	Leptocheirus plumulosus	Accredited	8/11/2008	NELAP	TX
2430	EPA 600-R-99-064	Chironomus tentans	Accredited	7/29/2008	NELAP	TX
2430	EPA 600-R-99-064	Hyalella azteca	Accredited	7/29/2008	NELAP	TX
2430	EPA 600-R-99-064	Lumbriculus variegatus	Accredited	7/29/2008	NELAP	TX
2431	ASTM E1706-05	Chironomus tentans	Accredited	7/29/2008	NELAP	TX
2431	ASTM E1706-05	Hyalella azteca	Accredited	7/29/2008	NELAP	TX
2432	ASTM E1688-00a	Corbicula fluminea	Accredited	7/29/2008	NELAP	TX
2432	ASTM E1688-00a	Macoma nasuta	Accredited	7/29/2008	NELAP	TX
2432	ASTM E1688-00a	Mercenaria mercenaria	Accredited	7/29/2008	NELAP	TX
2432	ASTM E1688-00a	Nereis virens	Accredited	7/29/2008	NELAP	TX
2433	ASTM E1367-03	Ampelisca abdita	Accredited	8/22/2008	NELAP	TX

Non-Potable Water Certification

Method Code	Method Ref	Analyte	Status	Date Effective	Type	AA
2146	EPA 2002.0/Freshwater Acute/EPA 821-R-02-012	Ceriodaphnia dubia	Accredited	7/29/2008	NELAP	TX
2147	EPA 2021.0/Freshwater Acute/ EPA 821-R-02-012	Daphnia magna	Accredited	7/29/2008	NELAP	TX
2147	EPA 2021.0/Freshwater Acute/ EPA 821-R-02-012	Daphnia pulex	Accredited	7/29/2008	NELAP	TX
2150	EPA 2000.0/Freshwater Acute/EPA 821-R-02-012	Pimephales promelas	Accredited	7/29/2008	NELAP	TX
2151	EPA 2007.0/Acute/EPA 821-R-02-012	Mysidopsis bahia	Accredited	7/29/2008	NELAP	TX
2152	EPA 2004.0/Acute/EPA 821-R-02-012	Cyprinodon variegatus	Accredited	7/29/2008	NELAP	TX
2153	EPA 2006.0/Acute/EPA 821-R-02-012	Menidia beryllina	Accredited	7/29/2008	NELAP	TX
2153	EPA 2006.0/Acute/EPA 821-R-02-012	Menidia menidia	Accredited	7/29/2008	NELAP	TX
10114600	EPA 1000	Pimephales promelas	Accredited	7/29/2008	NELAP	TX
10115001	EPA 1002	Ceriodaphnia dubia	Accredited	7/29/2008	NELAP	TX
10115205	EPA 1003	Selenastrum capricornutum	Accredited	8/11/2008	NELAP	TX
10115409	EPA 1004	Cyprinodon variegatus	Accredited	7/29/2008	NELAP	TX

Issue Date: August 22, 2008
Expiration Date: June 30, 2009

Print Date 8/22/2008 11:19:01 AM

Louisiana Environmental Laboratory
LELAP
Accreditation Program

Laboratory Scope of Accreditation

Page 2 of 2

Organization

04087 (713) 977-1500
PBS&J Environmental Toxicology Laboratory
888 West Sam Houston Pkwy., Suite 110
Houston, TX 77042-1917

Non-Potable Water Certification

Method Code	Method Ref	Analyte	Status	Date Effective	Type	AA
10115807	EPA 1006	Menidia beryllina	Accredited	7/29/2008	NELAP	TX
10115004	EPA 1007	Mysidopsis bahia	Accredited	7/29/2008	NELAP	TX

Issue Date: August 22, 2008
Expiration Date: June 30, 2009

Print Date 8/22/2008 11:19:01 AM

State of Florida
Department of Health, Bureau of Laboratories
This is to certify that

E871033

PBS&J ENVIRONMENTAL TOXICOLOGY LABORATORY
888 WEST SAM HOUSTON PARKWAY SOUTH, SUITE 110
HOUSTON, TX 77042

has complied with Florida Administrative Code 64E-1,
for the examination of Environmental samples in the following categories

NON-POTABLE WATER - TOXICITY, SOLID AND CHEMICAL MATERIALS - TOXICITY

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

EFFECTIVE July 01, 2008 THROUGH June 30, 2009

Max Seilinger, M.D.
Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04
NON-TRANSFERABLE E871033-02-07/01/2008
Supersedes all previously issued certificates

Charlie Crist
Governor

FLORIDA DEPARTMENT OF HEALTH

Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Page 1 of 2

Attachment to Certificate #: E871033-02, expiration date June 30, 2009. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E871033 **EPA Lab Code: TX01404** **(713) 977-1500**

E871033
PBS&J Environmental Toxicology Laboratory
888 West Sam Houston Parkway South, Suite 110
Houston, TX 77042

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Ceriodaphnia dubia	EPA 821-R-02-012 (FW acute)(2002.0)	Toxicity	NELAP	11/8/2007
Ceriodaphnia dubia	EPA 821-R-02-013 (FW chronic)(1002.0)	Toxicity	NELAP	11/8/2007
Cyprinella lecdsi	EPA 821-R-02-012 (FW acute)(2000.0)	Toxicity	NELAP	11/8/2007
Cyprinodon variegatus	EPA 821-R-02-012 (SW acute)(2004.0)	Toxicity	NELAP	11/8/2007
Cyprinodon variegatus	EPA 821-R-02-014 (SW chronic)(1004.0)	Toxicity	NELAP	11/8/2007
Daphnia magna	EPA 821-R-02-012 (FW acute)(2021.0)	Toxicity	NELAP	11/8/2007
Daphnia pulex	EPA 821-R-02-012 (FW acute)(2021.0)	Toxicity	NELAP	11/8/2007
Menidia beryllina	EPA 821-R-02-012 (SW acute)(2006.0)	Toxicity	NELAP	11/8/2007
Menidia beryllina	EPA 821-R-02-014 (SW chronic)(1006.0)	Toxicity	NELAP	11/8/2007
Menidia menidia	EPA 821-R-02-012 (SW acute)(2006.0)	Toxicity	NELAP	11/8/2007
Menidia peninsulae	EPA 821-R-02-012 (SW acute)(2006.0)	Toxicity	NELAP	11/8/2007
Mysidopsis bahia	EPA 821-R-02-012 (SW acute)(2007.0)	Toxicity	NELAP	11/8/2007
Mysidopsis bahia	EPA 821-R-02-014 (SW chronic)(1007.0)	Toxicity	NELAP	11/8/2007
Pimephales promelas	EPA 821-R-02-012 (FW acute)(2000.0)	Toxicity	NELAP	11/8/2007
Pimephales promelas	EPA 821-R-02-013 (FW chronic)(1000.0)	Toxicity	NELAP	11/8/2007
Selenastrum capricornutum	EPA 821-R-02-013 (FW chronic)(1003.0)	Toxicity	NELAP	11/8/2007

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2008** **Expiration Date: 6/30/2009**

Charlie Crist
Governor

FLORIDA DEPARTMENT OF HEALTH

Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

Page 2 of 2

Attachment to Certificate #: E871033-02, expiration date June 30, 2009. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E871033 **EPA Lab Code: TX01404** **(713) 977-1500**

E871033
PBS&J Environmental Toxicology Laboratory
888 West Sam Houston Parkway South, Suite 110
Houston, TX 77042

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Neteris viridis	EPA/823-B-98/004	Toxicity	NELAP	11/8/2007

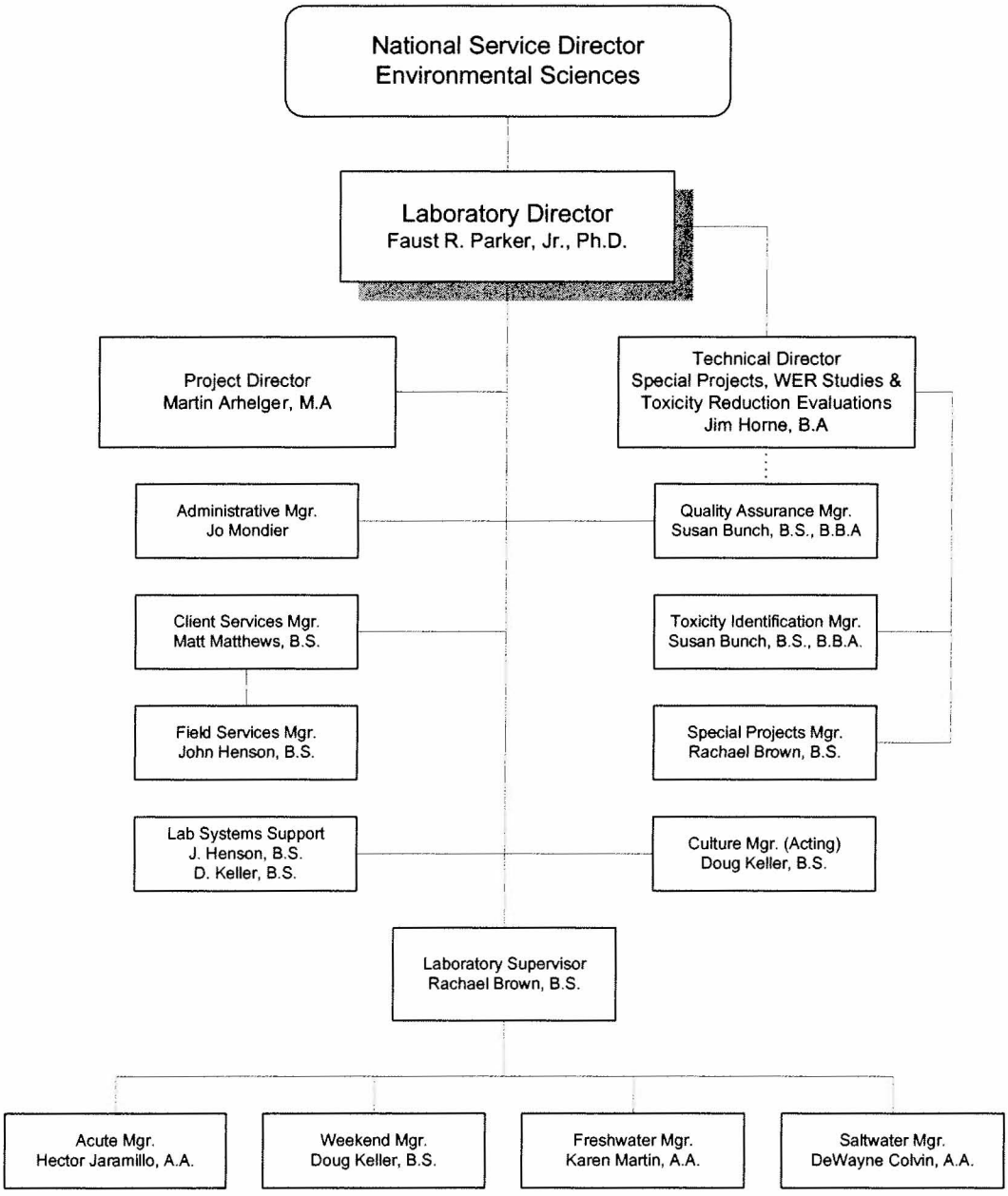
Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2008** **Expiration Date: 6/30/2009**

Appendix B - Key Staff Job Descriptions

Descriptions of additional staff positions, identified below, are maintained by the Quality Assurance Unit.

Acute Biomonitoring Manager
Administrative Assistant
Culture Manager
Culturist
Data Management Specialist
Field Services Manager
Field Services Technician
Freshwater Biomonitoring Manager
Health and Safety Officer
Lab Systems-Support Coordinator
Lab Technician
Saltwater Biomonitoring Manager
Senior Culture Biologist
Special Projects Director
Special Projects Manager
TRE Director
TRE Manager
Weekend Biomonitoring Manager

PBS&J Environmental Toxicology



PBS&J

Position Description

Laboratory Director

Summary

The Laboratory Director is responsible for overall operation of the organization including fiscal resources and personnel. The Director, acting within the limits of corporate authority, establishes laboratory policies and procedures and provides resources needed to effectively conduct the business of the laboratory including the Quality System.

Duties and Responsibilities

- Reviews and approves for distribution, work products resulting from services performed for clients
- Initiates and approves changes to the Quality System
- Reviews all non-conformance forms
- Prepares and controls the laboratory budget
- Prepares financial reports for regional management
- Oversees employee performance and salary evaluations
- Reports to the National Service Director - Environmental Sciences

Educational Requirements

Ph.D. in Physical or Biological Sciences, or a lesser degree with appropriate work experience and demonstrated performance abilities.

Experience/Skill Required

Ten years experience in environmental chemistry, toxicology, ecology or biology. Effective written and verbal communication skills. Strong management skills. PBS&J Project Management training completed.

_____ _____

Laboratory Director Date

Revised: August 24, 2007

PBS&J

Position Description

Technical Director

Summary

The Technical Director, acting within broad limits of authority delegated by the Division Manager/Laboratory Director, establishes standards of performance of the laboratory-- including development and implementation of the quality system and quality control practices and procedures--and monitors the validity of analyses conducted and data generated by the laboratory. Assists the Laboratory Director with day-to-day assessment of the laboratory's capabilities and participates in long-range planning and budgeting.

Duties and Responsibilities

- Establishes standard operating procedures (SOPs).
- Provides oversight and guidance to the Quality Assurance Unit.
- Reviews, and approves for distribution, work products resulting from services performed for clients.
- Prepares responses to requests for project proposals and cost quotations
- Develops new laboratory capabilities associated with agency requirements and new test methods
- Participates in Quality Board activities
- Reports to Laboratory Director

Educational Requirements

Ph.D. in Physical or Biological Sciences, or a lesser degree with appropriate work experience and demonstrated performance abilities.

Experience/Skill Required

Ten or more years of technical and management experience in environmental/aquatic toxicology. Participation in work-shops on federal or state protocols for aquatic toxicity evaluations and monitoring. Strong project management and supervisory skills. Effective written and verbal communication skills.

_____ _____
Technical Director Date

_____ _____
Laboratory Director Date

Revised: August 24, 2007

PBS&J

Position Description

Quality Assurance Officer

Summary

The Quality Assurance Officer (QAO) assures conformance to all quality requirements through competent management of the quality system. The QAO must have the ability and authority to recommend and implement immediate corrective measures and therefore is provided direct access to the highest level of management. The QAO must have a general knowledge of the analytical test methods for which data review is performed and be able to evaluate the data objectively. The QAO may be designated other duties, however these duties cannot bias the performance of the duties and responsibilities assigned to the QAO.

Duties and Responsibilities

- Performs Quality Systems and Operations Audits
- Reviews and maintains Personnel Qualification Records
- Oversees and coordinates training of all technical personnel
- Reviews data results for adherence to Data Quality Objectives
- Coordinates preparation of quality assurance reports to management, clients and regulatory agencies
- Reviews corrective action reports, recommends corrective action measures and monitors progress to closure
- Is knowledgeable in the Quality Systems as defined by NELAC
- Maintains, amends and distributes Quality Assurance Manuals, Standard Operating Procedures, Quality Assurance Procedures, and Quality Assurance Forms
- Reviews new or proposed protocols to determine appropriate use
- Coordinates and participates in Quality Board activities
- Reports to the Laboratory Director on a regular basis regarding the effectiveness of the Quality System

Educational Requirements

B.S. in physical or biological sciences required; M.S. preferred.

Experience/Skill Required

Substantial previous experience in environmental toxicology. Excellent written and communication skills. Strong collaborative project management skills. Must be a self starter and have the ability to work with little supervision.

_____ _____
Quality Assurance Officer Date

_____ _____
Laboratory Director Date

Revised: August 24, 2007

Position Description

Laboratory Supervisor

Summary

The Laboratory Supervisor is responsible for direct supervision of testing laboratory technical personnel and operations, ensuring adherence to laboratory procedures and accepted techniques. The individual is also responsible for assuring that test data recorded on data forms accurately documents test results. The Laboratory Supervisor reports directly to the Laboratory Director and provides direct supervision to test room managers and technical personnel.

Duties and Responsibilities

- Schedules and supervises technical personnel to meet test schedule requirements; coordinates staff assignments among test areas to achieve short-term workload/staff balance.
- In conjunction with Quality Assurance Manager, coordinates the training of testing laboratory technical personnel and ensures continued adherence to established protocols.
- Supervises the completeness-and-accuracy review of completed data sets.
- Participates in mid-year and annual review of supervised personnel.
- Participates in the hiring/discipline/dismissal of technical personnel to be supervised.
- Participates in Quality Board Activities.

Educational Requirements

B.S. in physical or biological sciences preferred; experience/aptitude may substitute for educational requirements.

Experience/Skill Required

Substantial previous experience as an environmental toxicology manager. Strong supervisory and communication skills.

_____ _____
Laboratory Supervisor Date

_____ _____
Laboratory Director Date

Revised: August 24, 2007

PBS&J

Position Description

Client Services Manger

Summary

The Client Services Manager acts as a liaison between the client and the organization. The individual coordinates project activities including budgeting, test scheduling and billing. The client services manager reviews project data prior to final report to assure adherence to specified project objectives.

Duties and Responsibilities

- Reviews and maintains client permit data and files
- Coordinates test schedules according to client permit specifications or client requests
- Prepares and updates permit summaries
- Maintains work project budgets including billing and invoicing
- Maintains client database system
- Prepares and negotiates work project quotes with clients
- Obtains contractual paperwork from clients
- Performs final review of report drafts
- Reports directly to the Laboratory Director

Educational Requirements

B. S. in physical or biological sciences.

Experience/Skill Required

Substantial previous experience in environmental toxicology. Excellent written and communication skills. Strong collaborative project management skills.

_____ _____

Client Services Manager Date

_____ _____

Laboratory Director Date

Revised: August 24, 2007

Volume 2
QUALITY ASSURANCE PROCEDURES

PBS&J
Environmental Toxicology Laboratory
888 West Sam Houston Parkway South, Suite 110
Houston, TX 77042-1917
Tel: 713-977-1500
Fax: 713-977-9233

Faust R. Parker, Jr., Ph.D.
Vice President & Division Manager
Laboratory Director

James D. Horne
Technical Director

Susan Bunch
Quality Assurance Manager

Rachael Brown
Laboratory Supervisor

Matt Matthews
Client Services Manager

Quality Assurance Procedures (QAPs) are designed to be reviewed and/or revised independent of each other; therefore, pagination within this Volume 2 of the Quality Assurance Manual is not sequential.

Q A P	ORGANIZATION AND MANAGEMENT	QAP No.	1
		Date:	03/26/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	M. Alejandra Garrido	A. Garrido	3/26/01
Quality Assurance Unit:	M. Alejandra Garrido	A. Garrido	3/26/01
Laboratory Director:	Faust R. Parker, Jr.	Faust R. Parker	3-27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Bund	9/25/03	Faust R. Parker	9-25-03	No changes	1.0
Susan Bund	9/8/05	Faust R. Parker	9-9-05	Updated to new data	2.0
Susan Bund	1/29/07	Faust R. Parker	1-29-07	No changes	2.0
Susan Bund	10/8/08	Faust R. Parker	10-8-08	No changes	2.0

QAP No. 1	Rev. No.: 2.0	Date: August 22, 2005

ORGANIZATION AND MANAGEMENT

1.0 PURPOSE

This Quality Assurance Policy provides a legal definition of the laboratory and its capabilities, the organization structure and the responsibilities of management

2.0 LEGAL DEFINITION OF THE LABORATORY

The laboratory is legally identifiable as:

Name of Firm: PBS&J Environmental Toxicology Laboratory

Address of Firm: 888 West Sam Houston Parkway South, Suite 110
Houston, TX 77042-1917

3.0 SCOPE

The Laboratory provides toxicity testing and consulting services to support wastewater discharge permit whole effluent toxicity and toxicity identification and reduction evaluations; marine and freshwater whole sediment toxicity tests and bioaccumulation assessments; and environmental fate and effects testing on industrial and consumer products, including drilling fluid systems and additives.

The laboratory is organized and operates in such a way as to meet the requirements of NELAC and to satisfy the needs of the client, the regulatory authorities, and organizations providing recognition. This pertains to work carried out in all laboratory facilities; whether on or off-sight, permanent or temporary.

4.0 Organization

The Laboratory:

A. Has managerial and technical personnel with the authority and resources needed to carry out their duties and to identify the occurrence of departures from the quality system or from the procedures for performing environmental tests, and to initiate actions to prevent or minimize such departures.

B. Has processes to ensure that its management and personnel are free from any undue internal and external commercial, financial and other pressures and influences that may adversely affect the quality of their work.

ORGANIZATION AND MANAGEMENT

C. Has policies and procedures to ensure the protection of its clients' confidential information and proprietary rights, including procedures for protecting the electronic storage and transmission of results.

D. Has policies and procedures to avoid involvement in any activities that would diminish confidence in its competence, impartiality, judgment or operational integrity.

E. Defines the organization and management structure of the laboratory, its place in the parent organization, and the relationships between quality management, technical operations and support services.

F. Specifies and documents the responsibility, authority, and interrelationships of all personnel who manage, perform or verify work affecting the quality of the environmental tests. Such documentation includes:

1) An organization chart with clear description of the lines of responsibility.

2) Job descriptions for all positions (maintained in personnel training files).

G. Provides supervision of environmental testing staff, including trainees, by persons familiar with the methods and procedures, purpose of each environmental test, and with the assessment of the environmental test results. The ratio of supervisory to non-supervisory personnel ensures adequate supervision for adherence to laboratory procedures and techniques.

H. Has technical management which has overall responsibility for the technical operations and the provision of the resources needed to ensure the required quality of laboratory operation.

The technical director:

1. Certifies that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited. Such certification shall be documented.

2. Shall meet the requirements specified in the Accreditation Process.

ORGANIZATION AND MANAGEMENT

I. Has a Quality Assurance Officer (QAO) who has the responsibility for the quality system and its implementation. The QAO has direct access to the highest level of management at which decisions are taken on laboratory policy or resources, and to the Technical Director

The QAO:

I. serves as the focal point for QA/QC and is responsible for the oversight and or review of quality control data;

II. has functions independent from laboratory operations for which the officer has quality assurance oversight;

III. is able to evaluate data objectively and perform assessments without outside (managerial) influence;

IV. has documented training and/or experience in QA/QC procedures and is knowledgeable in the quality system as defined under NELAC.

V. has a general knowledge of the analytical test methods for which data review is performed;

VI. conducts internal audits on the entire technical operation annually; and

VII. notifies the laboratory management of deficiencies in the quality system and monitors corrective action.

J. Appoints deputies for key managerial personnel, including the technical director(s) and/or quality-manager.

K. Participates in a proficiency test program as outlined in NELAC standards.

Q A P	QUALITY SYSTEM-ESTABLISHMENT	QAP No.	2
		Date:	03/06/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	M. Alejandra Garrido	A Garrido	3/26/01
Quality Assurance Unit:	M. Alejandra Garrido	A Garrido	3/26/01
Laboratory Director:	Faust R. Parker, Jr.	Faust R Parker Jr	3-27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Bunch	9/25/03	Faust Parker	9-25-03	No changes	1.0
Susan Bunch	9/8/05	Faust Parker	9-9-05	Updated - New Std's	2.0
Susan Bunch	1/29/07	Faust Parker	1-29-07	No changes	2.0
Susan Bunch	10/8/08	Faust R Parker	10-8-08	No changes	2.0

QAP No. 2	Rev. No.: 2.0	Date: August 31, 2004

QUALITY SYSTEM - ESTABLISHMENT

1.0 PURPOSE

The purpose of this policy is to describe the establishment of the quality system at the PBS&J Environmental Toxicology Laboratory.

2.0 ESTABLISHMENT OF THE QUALITY SYSTEM

The laboratory has established and maintains a quality system based on the required elements contained in NELAC, Chapter 5 - Quality Systems. The quality system is appropriate to the type, range, and volume of environmental testing performed at the laboratory. The laboratory documents its policies, systems, programs, procedures and instructions to the extent necessary to assure the quality of the environmental test results. The system's documentation is communicated to, understood by, available to, and implemented by the appropriate personnel.

A. The elements of the quality system are documented in the Laboratory's quality manual.

B. The overall quality system objectives are documented in the PBS&J Operating Philosophy and the Management Policy Statement which are included in the Laboratory's quality manual. The Philosophy and the Management Policy Statement are issued under the authority of the laboratory director and include:

1) management's commitment to good professional practice and to the quality of its environmental testings services to clients;

2) management's statement of the laboratory's standard of service;

3) the objectives of the quality system;

4) a requirement that all personnel familiarize themselves with the quality documentation and implement the policies and procedures in their work;

5) management's commitment to compliance with NELAC standards

C. The guidelines of the Quality System are established and documented in Quality Assurance Policies and Standard Operating Procedures, they are part of the Quality Manual.

D. The quality manual is maintained current under the responsibility of the quality assurance officer.

Q A P	THE QUALITY ASSURANCE MANUAL AND RELATED QUALITY ASSURANCE DOCUMENTS	QAP No.	3
		Date:	03/06/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	M. Alejandra Garrido	A. Garrido	3/26/01
Quality Assurance Unit:	M. Alejandra Garrido	A. Garrido	3/26/01
Laboratory Director:	Faust R. Parker, Jr.	Faust R Parker Jr	3-27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Bund	9/25/03	Faust R Parker Jr	9-25-03	No changes	1.0
Susan Bund	7/8/05	Faust R Parker Jr	9-5-05	SB No changes ~~Updated to current Ver.~~	1.0
Susan Bund	1/29/07	Faust R Parker Jr	1-29-07	No changes	1.0
Susan Bund	10/8/08	Faust R Parker Jr	10-8-08	No changes	1.0

QAP No. 3	Rev. No.: 1.0	Date: March 6, 2001

QUALITY SYSTEMS - THE QUALITY ASSURANCE MANUAL AND RELATED QUALITY ASSURANCE DOCUMENTS

1.0 PURPOSE

The purpose of this Quality Assurance Policy is to identify and describe the contents of the Quality Manual and related quality assurance documents.

2.0 The Quality Manual

The quality manual and related documentation, state the laboratory's policies and operational procedures established in order to meet the requirements of NELAC standards.

The Quality Manual lists on the title page:

- a document title
- the laboratory's full name and address
- the name, address, and telephone number of individuals responsible for the laboratory
- the name of the quality assurance officer
- the identification of all major organizational units which are covered by this quality manual and the effective date of the version

The quality manual and related quality documentation also contains:

a) quality policy statement, including objectives and commitments, by top management;

b) the organization and management structure of the laboratory, its place in PBS&J and relevant organizational charts;

c) the relationship between management, technical operations, support services and the quality system;

d) procedures to ensure that all records required under NELAC, Chapter 5 - Quality Systems are retained, as well as procedures for control and maintenance of documentation through a document control system which ensure that all SOPs, manuals, and documents clearly indicate the time period during which the procedure or document was in force;

e) job descriptions of key staff and reference to the job descriptions of other staff;

f) identification of the laboratory's approved signatories; the tittle page includes signed and dated concurrence of the QAO, technical director(s), and the laboratory director in charge of all laboratory activities (Laboratory Director);

g) the laboratory's procedures for achieving traceability of measurements;

h) a list of all test methods under which the laboratory performs its accredited testing;

i) mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work;

j) reference to the calibration and verification test procedures used;

k) procedures for handling submitted samples;

l) reference to the major equipment and reference measurement standards used as well as the facilities and services used in conducting tests;

m) reference to procedures for calibration, verification and maintenance of equipment;

n) reference to verification practices including interlaboratory comparisons, use of reference materials and internal quality control schemes;

o) procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur;

p) the laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications;

q) procedures for dealing with complaints;

r) procedures for protecting confidentiality and proprietary rights;

s) procedures for audits and data review;

t) process/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and are receiving any needed training;

u) process/procedures for educating and training personnel in their ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions;

v) reference to procedures for reporting analytical results; and,

w) a Table of Contents, and applicable lists of references and glossaries and appendices.

Q A P	QUALITY SYSTEMS AUDITS AND CORRECTIVE ACTIONS	QAP No.	4
		Date:	03/06/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	M. Alejandra Garrido	A. Garrido	3/26/01
Quality Assurance Unit:	M. Alejandra Garrido	A. Garrido	3/26/01
Laboratory Director:	Faust R. Parker, Jr.	Faust R Parker	3-27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Bund	9/25/03	Faust R Parker	9-25-03	No changes	1.0
Susan Bund	9/8/05	Faust R Parker	9-9-05	Updated - current Stds	2.0
Susan Bund	1/29/07	Faust R Parker	1-29-07	No Changes	2.0
Susan Bund	10/8/08	Faust R Parker	10-8-08	No changes	2.0

QAP No. 4	Rev. No.: 2.0	Date: August 31, 2005

QUALITY SYSTEMS - Audits and Corrective Actions

1.0 PURPOSE

The purpose of this Quality Assurance Policy is to describe the laboratory's internal audits procedures and the ongoing verifications to ensure that operations continue to conform to the requirements of the quality system.

2.0 INTERNAL AUDITS

A) The laboratory arranges for annual internal audits to verify that its operations continue to comply with the requirements of the laboratory's quality system and NELAC Standards.

B) The internal audit program shall address all elements of the quality system, including the environmental testing activities.

C) The Quality Assurance Officer (QAO) is responsible for planning and organizing audits.

D) The QAO conducts audits without outside influence and is independent of the activity to be audited.

E) Personnel do not audit their own activities.

F) Where audit findings cast a doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's environmental test results, the laboratory takes timely corrective action, and notifies clients in writing if investigations show that the laboratory results may have been affected.

3.0 MANAGERIAL REVIEW - The Quality Board

A) Laboratory Management conducts reviews of its quality systems and its testing and calibration activities through a **Quality Board**. The Quality Board was established to ensure the continuing suitability and effectiveness of the laboratory's quality system and to introduce any necessary changes or improvements.

B) The review by the Quality Board takes account of:

1) the suitability of policies and procedures;

2) reports from managerial and supervisory personnel;

3) the outcome of recent internal audits;

4) corrective and preventive actions;

5) assessments by external bodies

6) the results of interlaboratory comparisons;

7) any changes in the volume and type of work undertaken;

8) feedback from clients;

9) complaints;

10) other relevant factors.

C) The laboratory has a procedure for review by the Quality Board and maintains records of review findings and actions.

4.0 AUDIT REVIEW

A) All audit review findings and corrective actions that arise from them shall be documented.

B) The Laboratory Management shall ensure that these actions are discharged within the agreed time frame.

C) Follow-up audit activities shall verify and record the implementation and effectiveness of the corrective action taken.

5.0 PERFORMANCE AUDITS

In addition to quality audits the laboratory shall ensure the quality of results provided to clients by implementing checks to monitor the quality of the laboratory's analytical activities. Examples of these checks are:

A) internal quality control procedures;

B) participation in interlaboratory comparisons/proficiency testing;

C) use of certified reference materials;

D) replicate testing using the same or different test methods;

6.0 CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for corrective actions in SOPs, the laboratory has implemented a general procedure to be followed to determine when departures from documented policies, procedures and quality control have occurred. The procedure:

A) identifies the individuals responsible for assessing each QC data type;

B) identifies the individual responsible for initiating and/or recommending corrective actions;

C) defines how the analyst shall treat a data set if the associated QC measurements are unacceptable;

D) defines how out-of -control situations and subsequent corrective actions are to be documented; and,

E) specifies procedures for management (including the QAO) to review corrective action reports.

Q A P	QUALITY SYSTEMS - ESSENTIAL QUALITY CONTROL PROCEDURES	QAP No.	5
		Date:	03/06/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	M. Alejandra Garrido	A. Garrido	3/26/01
Quality Assurance Unit:	M. Alejandra Garrido	A. Garrido	3/26/01
Laboratory Director:	Faust R. Parker, Jr.	Faust R Parker Jr	3-27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Bunel	9/25/03	Faust R Parker Jr	9-25-03	No changes	1.0
Susan Bunel	9/8/05	Faust R Parker Jr	9-9-05	No changes	1.0
Susan Bunel	1/29/07	Faust R Parker Jr	1-29-07	No changes	1.0
Susan Bunel	10/8/08	Faust R Parker Jr	10-8-08	No changes	1.0

QAP No. 5	Rev. No.: 1.0	Date: March 6, 2001

QUALITY SYSTEMS - Essential Quality Control Procedures

1.0 PURPOSE

The purpose of this Quality Assurance Procedure is to identify and describe the essential quality control procedures at the PBS&J Environmental Toxicology Laboratory.

2.0 ESSENTIAL QUALITY CONTROL PROCEDURES

A) The laboratory has protocols in place to monitor the following quality controls:

1) Adequate positive and negative controls to monitor tests such as blanks and a reference toxicant program;

2) Adequate tests to define the variability and or repeatability of the laboratory results (replicates);

3) Measures to assure the accuracy of the test method including sufficient calibration, continuing calibration, and proficiency test samples;

4) Selection of appropriate formulae to reduce raw data to final results;

5) Selection and use of reagents and standards of appropriate quality;

6) Measures to assure constant and consistent test conditions (temperature and light);

B) All quality control measures shall be assessed and evaluated on an on-going basis, and quality control acceptance criteria shall be used to determine the usability of the data.

C) The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exist.

D) The quality control protocols specified by the laboratory's method manual shall be followed.

Q A P	PERSONNEL	QAP No.	6
		Date:	03/06/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	M. Alejandra Garrido	A. Garrido	3/26/01
Quality Assurance Unit:	M. Alejandra Garrido	A. Garrido	3/26/01
Laboratory Director:	Faust R. Parker, Jr.	Faust R. Parker, Jr.	3-27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Oscar Bunol	9/25/03	Faust R. Parker, Jr.	9-25-03	No changes	1.0
Oscar Bunol	9/8/05	Faust R. Parker, Jr.	9-5-05	Updated	2.0
Oscar Bunol	11/29/07	Faust R. Parker, Jr.	1-29-07	No changes	2.0
Oscar Bunol	10/8/08	Faust R. Parker, Jr.	10-8-08	No changes	2.0

QAP No. 6	Rev. No.: 2.0	Date: August 31, 2005

PERSONNEL

1.0 PURPOSE

The purpose of this Quality Assurance Procedure is to identify and describe the general requirements for laboratory staff, management responsibility and personnel record retention.

2.0 GENERAL REQUIREMENTS FOR LABORATORY STAFF

A) Laboratory management shall ensure the competence of all who operate specific equipment, perform environmental tests, evaluate results, and sign test reports.

B) Appropriate supervision is provided for employees undergoing training.

C) Personnel performing specific tasks are qualified on the basis of appropriate education, training, experience, and/or demonstrated skills.

D) The laboratory shall have sufficient personnel with the necessary education, training, technical knowledge, and experience for their assigned functions.

E) All personnel shall be responsible for complying with all quality assurance, quality control requirements that pertain to their organizational or technical function.

F) Each technical staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular function.

G) Each technical staff member must have a combination of experience and education to adequately demonstrate a general knowledge of the test methods, quality assurance, quality control procedures and records management.

H) Management shall formulate the goals with respect to the education, training, and skills of personnel. The laboratory shall have a policy and procedures for identifying training needs and providing training of personnel, relevant to the present and anticipated tasks of the laboratory.

I) The laboratory shall use personnel who are employed by, or under contract to, the laboratory. Where contracted or additional technical and key support personnel are used, the laboratory shall ensure that such personnel are supervised and competent and that they work in accordance with the laboratory's quality system.

J) The laboratory shall maintain current job descriptions for all personnel who manage, perform, or verify work affecting the quality of the environmental tests.

K) Management shall authorize specific personnel to perform particular types of sampling, environmental testing, to issue test reports, to give opinions and interpretations and to operate particular types of equipment. The laboratory shall maintain records of the relevant authorizations(s), competence, educational and professional qualification, training, skills and experience of all technical personnel, including contracted personnel. This information shall be readily available and shall include the date on which authorization and/or competence is confirmed.

L) Records on the relevant qualifications, training, skills, and experience of the technical personnel shall be maintained by the laboratory, including records on demonstrated proficiency for each test method.

3.0 LABORATORY MANAGEMENT RESPONSIBILITIES

The Laboratory Management shall be responsible for:

A) defining the minimal level of qualification, experience and skills necessary for all positions in the laboratory;

B) ensuring that all technical laboratory staff have demonstrated capability in the activities for which they are responsible;

C) ensuring that the training of each member of the technical staff is kept up-to-date by the following:

1) Evidence must be on file that demonstrates that each employee has read, understood, and is using the latest version of the laboratory's in-house quality documentation, which relates to his/her job responsibilities.

2) Training courses or workshops on specific equipment, analytical techniques or laboratory procedures shall all be documented.

3) Analyst training shall be considered up to date if an employee's training file contains a certification that he/she has read, understood and agreed to perform the most recent version of the test method; and documentation of continued proficiency once per year.

D) documenting all analytical and operational activities of the laboratory;

E) supervising all personnel employed by the laboratory;

F) ensuring that all sample acceptance criteria are verified and that samples are logged into the sample tracking system and properly labeled and stored;

G) documenting the quality of all data reported by the laboratory;

H) developing a pro-active program for prevention and detection of improper, unethical or illegal actions;

4.0 RECORDS

A) Records on the relevant qualifications, training skills and experience of the technical personnel shall be maintained by the laboratory.

B) Records on demonstrated proficiency shall be maintained by the laboratory.

Q A P	PHYSICAL FACILITIES - ACCOMMODATION AND ENVIRONMENT	QAP No.	7
		Date:	03/06/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	M. Alejandra Garrido	A. Garrido	3/26/01
Quality Assurance Unit:	M. Alejandra Garrido	A. Garrido	3/26/01
Laboratory Director:	Faust R. Parker, Jr.	Faust R. Parker	3-27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Bund	9/25/03	Faust R. Parker	9-25-03	No changes	1.0
Susan Bund	9/8/05	Faust R. Parker	9-9-05	No changes	1.0
Susan Bund	1/29/07	Faust R. Parker	1-29-07	No changes	1.0
Susan Bund	10/8/08	Faust R. Parker	10-8-08	No changes	1.0

QAP No. 7	Rev. No.: 1.0	Date: March 6, 2001

PHYSICAL FACILITIES - ACCOMMODATION AND ENVIRONMENT

1.0 PURPOSE

The purpose of this Quality Assurance Procedure is to describe the laboratory's standards regarding its physical facilities.

2.0 ENVIRONMENT

A) Laboratory accommodation, test areas, energy sources, lighting, heating and ventilation shall be such as to facilitate proper performance of tests.

B) The environment in which these activities are undertaken shall not invalidate the results or adversely affect the required accuracy of measurement.

C) The laboratory shall provide for the effective monitoring, control and recording of environmental conditions as appropriate (lighting, temperature).

D) The laboratory shall document and adhere to the above mentioned when specified in a test method or by regulation.

3.0 WORK AREAS

A) There shall be effective separation between neighboring areas when the activities therein are incompatible including culture handling.

B) Access to and use of all areas affecting the quality of these activities shall be defined and controlled.

C) Adequate measures shall be taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality.

D) Work spaces must be available to ensure an unencumbered work area. Work areas include:

1) access and entryways to the laboratory
2) sample receipt area
3) sample storage area
4) chemical storage area
5) data handling and storage area

Q A P	EQUIPMENT, REFERENCE MATERIALS, MEASUREMENT TRACEABILITY AND CALIBRATION	QAP No.	8
		Date:	03/06/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	M. Alejandra Garrido	A. Garrido	3/26/01
Quality Assurance Unit:	M. Alejandra Garrido	A. Garrido	3/26/01
Laboratory Director:	Faust R. Parker, Jr.	Faust R. Parker	3-27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Bunch	4/29/03	Faust R. Parker	5-2-03	Revised 6.0, D	1.1
Susan Bunch	9/25/03	Faust R. Parker	9-26-03	No changes	1.1
Susan Bunch	9/8/05	Faust R. Parker	9-9-05	Updated	2.0
Susan Bunch	1/29/07	Faust R. Parker	1-29-07	No Changes	2.0
Susan Bunch	10/8/08	Faust R. Parker	10-8-08	No changes	2.0

QAP No. 8	Rev. No.: 2.0	Date: August 31, 2005

Equipment and Calibration

1.0 PURPOSE

This Quality Assurance Procedure details the requirements for the control of inspection, measuring, and testing equipment, support equipment and reference standards used at the PBS&J Environmental Toxicology Laboratory.

2.0 GENERAL REQUIREMENTS

A) The laboratory shall be furnished with all items of sampling, measurement, and test equipment required for proper environmental testing. In those cases where the laboratory needs to use equipment outside its permanent control, it shall ensure that the equipment and its use meets NELAC standards.

B) Equipment used for testing and sampling shall be capable of achieving the accuracy required and shall comply with specifications relevant to the environmental tests concerned. Before being placed into service, equipment (including that used for sampling) shall be calibrated or checked to establish that it meets the laboratory's specification requirements and complies with the relevant standard specifications.

3.0 SUPPORT EQUIPMENT

Support equipment refers to devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, thermometers, and volumetric dispensing devices (if quantitative results are dependent on their accuracy).

A) All support equipment shall be maintained in proper working order. The records of all repair and maintenance activities including service calls, shall be kept.

B) All support equipment shall be calibrated or verified at least annually, using NIST traceable references when available, over the entire range of use. The results of such calibration or verification shall be within the specifications of the application for which the equipment is used, or:

1) the equipment shall be removed from service until repaired; or

2) the laboratory shall maintain records of established correction factors to correct all measurements

C) Raw data records shall be retained to document equipment performance.

D) Prior to use on each working day, balances, ovens, and sample storage areas shall be checked in the expected use range, with NIST traceable references where commercially available. The acceptability for use or continued use shall be according to the needs of the analysis or application for which the equipment is being used.

MEASUREMENT SYSTEMS

A) PBS&J maintains a Measurement and Calibration System which assures that inspection, measuring and test equipment (devices used to gauge, measure, inspect, test or otherwise assess or assure conformance of materials) conform to appropriate specifications.

E) The Measurement and Calibration System is based on guidelines provided by:

1) Manufacturer guidelines and methods.
2) EPA and APHA Standard Methods

3.0 PROCEDURES

A) All equipment and standards requiring calibration are calibrated, used, and maintained in accordance with an approved standard operating procedure (SOP).

B) The Quality Assurance Unit (QAU) develops and maintains Standard Operating Procedures (SOPs) for the calibration of each piece of equipment. These techniques may be a combination of published standard practices, manufacturer's instructions or applicable portions thereof.

C) The QAU is responsible for identifying and providing suitable equipment to ensure contractual requirements are achieved.

4.0 CALIBRATION INTERVALS

A) Calibration intervals are established in the SOP for each type of measuring and test equipment. The calibration interval will be based on stability, application and degree of usage for each piece of equipment.

B) Established calibration intervals are adjusted where prior calibration results indicate this action is warranted. Measuring and test equipment are re-calibrated and/or serviced earlier than established intervals when they are damaged or performance is subject for any reason.

5.0 CALIBRATION SOURCES & DATA

A) All measuring and test equipment is calibrated against a standard whose calibration is certified as traceable to the National Institute of Standards and Technology (NIST) or equivalent. If standards are not available at NIST, industry standards will be used.

B) A standard may be calibrated by a qualified commercial or Government laboratory/agency.

C) The accuracy of each standard shall be supported by a certificate, a report or data which will be available upon request.

6.0 TRACEABILITY OF CALIBRATION

A) A calibration history is maintained for measuring and test equipment.

B) Meters used on a daily basis have their own calibration log books which are updated by laboratory personnel and periodically inspected and initialed by the QAU.

C) The QAU reviews all laboratory calibration logs.

D) Balances, and other equipment which require annual calibration, are certified by a contracted company. A certificate of calibration is issued by the company, which is archived by the QAU, and the equipment is labeled with a due date for recalibration.

7.0 DOCUMENTATION

A) Records shall be maintained of each major item of equipment and all reference materials significant to the tests performed.

B) These records shall include documentation on all routine and non-routine maintenance activities and reference material verifications.

C) The records shall include:

1) the name of the item of equipment;
2) the manufacturer's name, type identification, serial number;
3) date received and date place in service;
4) current location;
5) condition when received (new, used);
6) manufacture's instructions;
7) dates and results of calibration and verification and date of next calibration;
8) details of maintenance carried out to date;
9) history of damage, malfunction, repair.

8.0 RECALL SYSTEM

A schedule is established and monitored by the QAU which effectively assures that measuring and test equipment is re-calibrated on schedule or discontinued from use. The schedule is defined for each equipment item in the appropriate SOPs.

9.0 OUT-OF-CALIBRATION

A) When measuring, inspection, or testing equipment is found to be out-of calibration and not capable of being re-calibrated to the appropriate reference standard(s), a Non-Conformance Report (NCR) is prepared and submitted to the Laboratory Director and the QAU. The equipment item is clearly marked "Out-of-Service."

B) Out-of-calibration equipment will be quarantined by the QAU. The equipment will be repaired/adjusted, re-calibrated or replaced.

C) The QAU and the Laboratory Supervisor will evaluate the equipment fault condition(s) addressed by the NCR and initiate corrective action necessary to resolve any identified data quality issues.

Q A P	TEST METHODS AND STANDARD OPERATING PROCEDURES	QAP No.	9
		Date:	03/06/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	M. Alejandra Garrido	A. Garrido	3/26/01
Quality Assurance Unit:	M. Alejandra Garrido	A. Garrido	3/26/01
Laboratory Director:	Faust R. Parker, Jr.	Faust R Parker	3.27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Burch	9/25/03	Faust R Parker	9-26-03	Procedural Clarification	1.1
Susan Burch	9/8/05	Faust R Parker	9-9-05	Updated	2.0
Susan Burch	1/29/07	Faust R Parker	1-29-07	No changes	2.0
Susan Burch	10/8/08	Faust R Parker	10-8-08	No changes	2.0

QAP No. 9	Rev. No.: 2.0	Date: September 1, 2005

TEST METHODS AND STANDARD OPERATING PROCEDURES

1.0 PURPOSE

The purpose of this Quality Assurance Procedure is to identify and describe methods documentation and requirements of the laboratories Standard Operating Procedures.

2.0 METHODS DOCUMENTATION

A) The laboratory shall use appropriate methods and procedures for all environmental tests within its scope. These include sampling, handling, transport, storage, and preparation of samples, and, where appropriate, an estimation of the measurement uncertainty as well as statistical techniques for analysis of environmental test data.

B) The laboratory shall have instructions on the use and operation of all relevant equipment, and on the handling and preparation of samples where the absence of such instructions could jeopardize the results of environmental tests. All instructions, standards, manuals, and reference data relevant to the work of the laboratory shall be kept up to date and shall be made readily available to personnel. Deviation from environmental test methods shall occur only if the deviation has been documented, technically justified, authorized, and accepted by the client.

3.0 STANDARD OPERATING PROCEDURES (SOPs)

The Laboratory shall maintain SOPs that accurately reflect all phases of current laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, and all test methods.

A) These documents include equipment manuals provided by the manufacturer, or internally written documents.

B) The test methods may be copies of published methods as long as changes in the methods are documented and included in the methods manual.

C) Copies of SOPs shall be accessible to all personnel.

D) The SOPs shall be organized.

E) Each SOP shall clearly indicate the effective date of the document, the revision number and the signatures of the originator, the Quality Assurance Officer and the Laboratory Director.

F) SOPs and test methods shall be kept in a methods manual and/or the laboratory computer system.

4.0 TEST METHODS MANUALS

A) The laboratory shall have and maintain an in-house methods manual(s) for each accredited analyte or test method.

B) The manual may consist of copies of published or reference test methods or SOPs that have been written by the laboratory.

5.0 SOURCES OF METHODS

A) Methods published in international, regional, or national standards shall preferable be used. The laboratory shall ensure that it uses the latest valid edition of a standard unless it is not appropriate or possible to do so. When necessary, the standard shall be supplemented with additional details to ensure consistent application.

B) When the use of specific methods for testing are mandated or requested, only those methods shall be used.

C) The introduction of environmental test methods developed by the laboratory for its own use shall be a planned activity and shall be assigned to qualified personnel equipped with adequate resources.

D) Where test methods are employed that are not required, as in the PBMS approach, the methods shall be fully documented and validated, and be available to the client and other recipients of the relevant reports.

6.0 DEMONSTRATION OF CAPABILITY

The laboratory shall have a program and documentation of initial and continued demonstration of capability for each analyst.

7.0 DATA VERIFICATION

A) The laboratory shall establish SOPs to ensure that the reported data are free from transcription and calculation errors.

B) The laboratory shall establish SOPs to ensure that all quality control measures are reviewed and evaluated before data are reported.

C) The laboratory shall establish SOPs addressing manual calculations including manual integrations.

8.0 COMPUTERS AND ELECTRONIC DATA RELATED REQUIREMENTS

The laboratory shall ensure that computers used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data,:

A) all requirements of NELAC Chapter 5- Quality Systems are met;

B) computer software is documented and adequate for use;

C) procedures are established for protecting the integrity of data; such procedures shall include, but not be limited to, integrity of data entry or capture, data storage, data transmission and data processing;

D) computer and automated equipment are maintained to ensure proper functioning and provided with the environmental and operating conditions necessary to maintain the integrity of calibration and test data; and,

E) it establishes and implements appropriate procedures for the maintenance of security of data including the prevention of unauthorized amendment of, computer records.

Q A P	SAMPLE HANDLING, SAMPLE ACCEPTANCE AND SAMPLE RECEIPT	QAP No.	10
		Date:	03/06/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	Derek Beimgraben	Derek Beimgraben	3/27/01
Quality Assurance Unit:	M. Alejandra Garrido	A. Garrido	3/26/01
Laboratory Director:	Faust R. Parker, Jr.	Faust R. Parker, Jr.	3-27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Bunch	4/29/03	Faust R. Parker, Jr.	5-2-03	Revised S.O	1.1
Susan Bunch	9/25/03	Faust R. Parker, Jr.	9-25-03	No changes	1.1
Susan Bunch	9/8/05	Faust R. Parker, Jr.	9-9-05	updated	2.0
Susan Bunch	1/29/07	Faust R. Parker, Jr.	1-29-07	No changes	2.0
Susan Bunch	10/8/08	Faust R. Parker, Jr.	10-8-08	No changes	2.0

QAP No. 10	Rev. No.: 2.0	Date: September 8, 2005

SAMPLE HANDLING, SAMPLE ACCEPTANCE AND SAMPLE RECEIPT

1.0 PURPOSE

This Quality Assurance Procedure describes the laboratory's guidelines for sample handling, acceptance and receipt.

2.0 SAMPLE HANDLING

While the laboratory may not have control of field sampling activities, the following are essential to ensure the validity of the laboratory's data.

A) The laboratory shall have procedures for the transportation, receipt, handling, protection, storage, retention, and/or disposal of samples, including all provisions necessary to protect the integrity of the sample, and to protect the interests of the laboratory and the client.

B) The laboratory shall have a system for identifying samples. The identification shall be retained throughout the life of the sample in the laboratory. The system shall be designed and operated so as to ensure that samples cannot be confused physically or when referred to in records or other documents.

1) The laboratory shall assign a unique ID code to each sample container received in the laboratory.

2) The laboratory code shall maintain an unequivocal link with the unique field ID code assigned each container.

3) The laboratory ID code shall be placed on the sample container as a durable label.

4) The laboratory ID code shall be entered into the laboratory records and shall be the link that associated the sample with related laboratory activities such as sample preparation.

3.0 SAMPLE RECEIPT PROTOCOLS

A) Upon receipt, the temperature of the sample will be checked and recorded. Samples which require thermal preservation shall be considered acceptable if the arrival temperature is within 2°C of the required or method specified range. Samples that are hand delivered to the laboratory on the same day that they are collected may not meet these criteria. In these cases, the samples shall be considered

acceptable if there is evidence that the chilling process has begun such as arrival on ice.

B) When there is doubt as to the suitability of a sample for environmental testing, or when a sample does not conform to the description provided, or the test required is not specified in sufficient detail, the laboratory shall consult the client for further instructions before proceeding and shall record the discussion.

C) Each sample is uniquely identified with indelible ink on the sample container and the chain of custody.

D) Each sample received is documented on the sample receipt log. The sample receipt log shall record the following:

1) client/project name
2) date and time of laboratory receipt
3) unique laboratory ID code
4) initials of person receiving the sample

E) Any comments resulting from inspection of sample upon arrival shall be recorded on the accompanying chain of custody. A copy of the chain of custody is kept in a binder in the receiving area.

4.0 SAMPLE ACCEPTANCE POLICY

A) Each sample is required to have a chain of custody associated with it which will include the following information:

1) Client name, location and time of collection.

2) Collector's name.

3) Sample arrival date and time.

4) Signature of the person that checks in the sample.

5) Sample arrival temperature.

6) Sample type.

B) Each sample container must be labeled with the client name, location, and time of collection using indelible ink.

C) Only the use of approved containers are acceptable.

D) Samples must be used within the sample expiration times.

E) Sufficient sample volume must be available to perform the necessary tests.

F) Certain procedures must be followed when a sample shows signs of damage, contamination or inadequate preservation.

5.0 STORAGE CONDITIONS

Samples submitted for WET testing are stored in a walk - in cooler and maintained at 1 - 6°C when not in use. The samples are stored away from all standards, reagents, food, and other potentially contaminating sources, and in such a manner as to prevent cross contamination.

6.0 SAMPLE DISPOSAL

All sample disposition is carried out and recorded according to the corresponding SOP.

Q A P	RECORDS	QAP No.	11
		Date:	03/06/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	M. Alejandra Garrido	A. Garrido	3/26/01
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Laboratory Director:	Faust R. Parker, Jr.	Faust R. Parker	3-27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Bunch	10/23/02	Faust R. Parker	10-28-02	Revised 3.0 B	1.1
Susan Bunch	9/25/03	Faust R. Parker	9-26-03	No changes	1.1
Susan Bunch	9/8/05	Faust R. Parker	9-8-05	Updated	2.0
Susan Bunch	1/29/07	Faust R. Parker	1-29-07	No changes	2.0
Susan Bunch	10/8/08	Faust R. Parker	10-8-08	No changes	2.0

QAP No. 11	Rev. No.: 2.0	Date: September 1, 2005

RECORDS

1.0 PURPOSE

The purpose of this Quality Assurance Procedure is to identify and describe the record keeping system at the PBS&J Environmental Toxicology Laboratory.

2.0 RECORD KEEPING SYSTEM AND DESIGN

The record keeping system must allow historical reconstruction of all laboratory activities that produced the resultant sample analytical data. The history of the sample must be readily understood through the documentation. This shall include interlaboratory transfers of samples.

A) The records shall include the identity of personnel involved in sampling, preparation, calibration or testing.

B) All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification shall be documented.

C) The record keeping system shall facilitate the retrieval of all working files and archived records for inspection and verification purposes.

D) All documentation entries shall be signed or initialed by responsible staff. The reason for the signature or initials shall be clearly indicated in the records (such as "sampled by", prepared by" or "reviewed by").

E) All generated data except those that are generated by automated data collection systems, shall be recorded, directly, promptly and legibly in permanent ink.

F) Entries in records shall not be obliterated by methods such as erasures, overwritten files or markings. All corrections to record keeping errors shall be made by one line marked through the error. The individual making the correction shall initial and date the correction. These criteria also shall apply to electronically maintained records.

3.0 RECORDS MANAGEMENT AND STORAGE

A) All records (including those pertaining to calibration and test equipment), certificates and reports shall be safely stored, held secure and in confidence to the client. NELAP related records shall also be available to the accrediting authority.

B) All records shall be retained for five years from date of last use. All information necessary for the historical reconstruction of data must be maintained by the laboratory. After the five year retention period, documents are taken to a local recycling facility. All personnel have the authority to dispose of documents after the retention requirement has been met. Records which are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

C) Records that are stored or generated by computers or personal computers shall have hard copy or write-protected backup copies.

D) The laboratory shall establish a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation storage and reporting.

E) Access to archived information shall be documented with an access log. These records shall be protected against fire, theft, loss, environmental deterioration and, in the case of electronic records, electronic or magnetic sources.

F) In the event that the laboratory transfers ownership or goes out of business, all clients shall be contacted in order to establish procedures for transferring or disposing of their records. In the event that certain clients cannot be contacted, their records shall be retained by the parent company (PBS&J) for the period designated in the corporate policy manual.

4.0 LABORATORY SAMPLE TRACKING

A record of all procedures to which a sample is subjected while in the possession of the laboratory shall be maintained. These shall include but are not limited to all records pertaining to:

A) Sample preservation including appropriateness of sample container and compliance with holding time requirement;

B) Sample identification, receipt, acceptance or rejection and log-in;

C) Sample storage and tracking including shipping receipts, transmittal forms, and internal routing and assignment records;

D) Documented procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

5.0 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following shall be retained:

A) All original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts work sheets.

B) A written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;

C) Copies of final reports;

D) Archived SOPS;

E) Correspondence relating to laboratory activities for a specific project;

F) All corrective action reports, audits and audit responses;

G) Proficiency test results and raw data; and,

H) Data review and cross checking.

RECORDS

6.0 ANALYTICAL RECORDS

The essential information to be associated with analysis shall include:

A) Laboratory sample ID code;

B) Date and time of analysis;

C) Instrumentation identification and instrument operating conditions/parameters;

D) Analysis type;

E) All manual calculations; and,

F) Analysts's or operators initials/signatures.

G) Sample preparation including volumes, weights, meter readings, calculations, reagents;

H) Sample analysis;

I) Standard and reagent origin, receipt, preparation and use;

J) Calibration criteria, frequency and acceptance criteria;

K) Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

L) Quality control protocols and assessment

M) Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries;

N) Method performance criteria including expected quality control requirements.

7.0 ADMINISTRATIVE RECORDS

The following shall be maintained:

A) Personnel qualifications, experience and training records;

B) Records of Demonstration of Capability for each analyst; and

C) A log of names, individuals and signatures for all individuals responsible for signing or initialing any laboratory record.

Q A P	EVIDENTIARY CUSTODY AND DOCUMENTATION	QAP No.	12
		Date:	03/06/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	M. Alejandra Garrido	A. Garrido	3/26/01
Quality Assurance Unit:	M. Alejandra Garrido	A. Garrido	3/26/01
Laboratory Director:	Faust R. Parker, Jr.	Faust R. Parker	3-27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Bund	9/25/03	Faust R. Parker	9-26-03	No changes	1.0
Susan Bund	9/8/05	Faust R. Parker	9-9-05	no changes	1.0
Susan Bund	1/29/07	Faust R. Parker	1-29-07	No changes	1.0
Susan Bund	10/8/08	Faust R. Parker	10-8-08	No changes	1.0

QAP No. 12	Rev. No.: 1.0	Date: March 6, 2001

EVIDENTIARY CUSTODY AND DOCUMENTATION

1.0 PURPOSE

The purpose of this Quality Assurance Procedure is to describe the basic requirements of sample custody and required documentation.

2.0 BASIC REQUIREMENTS

Tracking records or chain of custody (COC) shall include by direct entry of linkage or linkage to other records:

A) Time of day and calendar date of each transfer or handling procedure;

B) Signatures of all personnel who handle the sample (collection, transfer, and receipt);

C) All information necessary to produce unequivocal, accurate records that document the laboratory activities associated with sample receipt, preparation, analysis and reporting; and

D) Common carrier document.

3.0 CONTROLLED ACCESS TO SAMPLES

The laboratory must be maintained as a secured area, restricted to authorized personnel only.

4.0 SAMPLE DISPOSAL

Records shall indicate the date of disposal, the nature of disposal and the name of the individual who performed the task.

Q A P	LABORATORY REPORT FORMAT AND CONTENTS	QAP No.	13
		Date:	03/06/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	M. Alejandra Garrido	A. Garrido	3/26/01
Quality Assurance Unit:	M. Alejandra Garrido	A. Garrido	3/26/01
Laboratory Director:	Faust R. Parker, Jr.	Faust R Parker	3-27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Bund	9/25/03	Faust R Parker	9-26-03	No changes	1.0
Susan Bund	9/8/05	Faust R Parker	9-8-05	No changes	1.0
Susan Bund	1/29/07	Faust R Parker	1-29-07	No changes	1.0
Susan Bund	10/8/08	Faust R Parker	10-8-08	No changes	1.0

QAP No. 13	Rev. No.: 1.0	Date: March 6, 2001

LABORATORY REPORT FORMAT AND CONTENTS

1.0 PURPOSE

The purpose of this Quality Assurance Procedure is to describe the format and contents of laboratory reports.

2.0 REQUIREMENTS

The results of all tests shall be recorded accurately, clearly, unambiguously and objectively. Test results shall be reported in a test report and shall include all the information necessary for the interpretation of the test results and all information required by the method used.

A) Each report to an outside client shall include:

1) a title;
2) the name and address of the laboratory and name and phone number of the contact person;
3) a job and document number to uniquely identify the report;
4) page numbers;
5) the name and address of the client, where appropriate the project name;
6) the sample identification code or number;
7) any deviations from prescribed requirements;
8) date of receipt of sample, date and time of sample collection, and holding time;
9) sampling procedure if sample was collected by the laboratory;
10) measurements and derived results and any failures identified;
11) identify whether data are calculated on a dry weight or wet weight basis;
12) identify the reporting units;
13) the statistical package used to provide data;
14) a signature and title of the person accepting responsibility for the content of the report;
15) clear identification of all test data provided by outside sources.

B) After issuance of the report, the report shall remain unchanged.

C) The laboratory shall notify clients in writing in any event such as the identification of defective measuring or test equipment that casts doubt on the validity of reported results.

D) Confidentiality shall be preserved in the event that test results are transmitted by telephone, telex, facsimile, or other electronic means.

Q A P	SUBCONTRACTING ANALYTICAL SAMPLES	QAP No.	14
		Date:	03/06/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	M. Alejandra Garrido	A. Garrido	3/26/01
Quality Assurance Unit:	M. Alejandra Garrido	A. Garrido	3/26/01
Laboratory Director:	Faust R. Parker, Jr.	Faust R Parker	3-27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Bunch	9/25/03	Faust R Parker	9-26-03	Clarification 2.0(c)	1.1
Susan Bunch	9/8/05	Faust R Parker	9-9-05	Updated	2.0
Susan Bunch	1/29/07	Faust R Parker	1-29-07	No changes	2.0
Susan Bunch	10/8/08	Faust R Parker	10-8-08	No changes	2.0

QAP No. 14	Rev. No.: 2.0	Date: August 31, 2005

SUBCONTRACTING ANALYTICAL SAMPLES

1.0 PURPOSE

The purpose of this Quality Assurance Procedure is to describe the guidelines to be followed when subcontracting services.

2.0 BASIC REQUIREMENTS

A) The laboratory shall subcontract work to a laboratory accredited under NELAP for the tests to be performed or with a laboratory that meets applicable statutory and regulatory requirements for performing the tests and submitting the results of tests performed.

B) The laboratory performing the subcontracted work shall be indicated in the final report and non-NELAP accredited work shall be clearly identified.

C) The laboratory shall advise the client of the arrangement in writing and, when possible, gain the approval of the client, preferably in writing.

D) The laboratory is responsible to the client for the subcontractor's work, except in the case where the client or a regulatory authority specifies which subcontractor is to be used.

E) The laboratory shall maintain a register of all subcontractors that it uses for environmental tests and a record of the evidence of compliance with section A) above.

Q A P	OUTSIDE SUPPORT SERVICES AND SUPPLIES	QAP No.	15
		Date:	03/06/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	M. Alejandra Garrido	A. Garrido	3/20/01
Quality Assurance Unit:	M. Alejandra Garrido	A. Garrido	3/26/01
Laboratory Director:	Faust R. Parker, Jr.	Faust R Parker	3-27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Bunal	7/25/03		9-26-03	No changes	1.0
Susan Bunal	9/8/05		9-9-05	No changes	1.0
Susan Bunal	1/29/07		1-27-07	No changes	1.0
Susan Bunal	10/8/08		10-8-08	No changes	1.0

QAP No. 15	Rev. No.: 1.0	Date: March 6, 2001

OUTSIDE SUPPORT SERVICES AND SUPPLIES

1.0 PURPOSE

The purpose of this Quality Assurance Procedure is to describe the laboratory's guidelines for obtaining outside support services and supplies.

2.0 BASIC REQUIREMENTS

A) Where the laboratory procures outside services and supplies in support of tests the laboratory shall use only those outside support services and supplies that are of adequate quality to sustain confidence in the laboratory's tests.

B) Where no independent assurance of the quality of outside support services or supplies is available, the laboratory shall have procedures to ensure that purchased equipment, materials and services comply with specified requirements.

C) The laboratory shall maintain records of all suppliers from whom it obtains support services or supplies required for tests.

Q A P	COMPLAINTS	QAP No.	16
		Date:	03/06/01

Origination and Acceptance:

	Name	Signature	Date
Originator:	M. Alejandra Garrido	A. Garrido	3/26/01
Quality Assurance Unit:	M. Alejandra Garrido	A. Garrido	3/26/01
Laboratory Director:	Faust R. Parker, Jr.	Faust R Parker Jr	3-27-01

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susa Bund	9/25/03	Faust R Parker Jr	9-26-03	No changes	1.0
Susa Bund	9/8/05	Faust R Parker	9-9-05	No changes	1.0
Susa Bund	1/29/07	Faust R Parker	1-29-07	No changes	1.0
Susa Bund	10/8/08	Faust R Parker	10-8-08	No changes	1.0

QAP No. 16	Rev. No.: 1.0	Date: March 6, 2001

COMPLAINTS

1.0 PURPOSE

The purpose of this Quality Assurance Procedure is to describe the laboratory's policy and procedure for the resolution of complaints received from clients or other parties about the laboratory's activities.

2.0 PROCEDURE

A) Client complaints may be handled by the Client Services Manager, Laboratory Director and/or Quality Assurance Manager.

B) An external NCR is completed in the same manner as those related to an internal non-conformance. The Laboratory Manager, Laboratory Director and Quality Assurance decide upon a Corrective Action, consult with the client as to its acceptability, and implement it.

C) The client is provided with a copy of the completed NCR, where required, client forms are used to document the process.

D) If a non-conforming report is shipped to a client, the Laboratory Manager will notify the client and initiate inspection of the report. If corrections are minor and limited to one or two pages corrections are made and the new pages, along with a letter of explanation, are issued to the client for insertion into the report. If major corrections are required the entire report is reissued, along with a letter of explanation.

E) Upon notification that a non-conforming report was received by a client, Quality Assurance will investigate and review the report during the correction process. Complaints concerning non-conforming reports shall be responded to expeditiously.

Q A P	COORDINATION OF QUALITY CONTROL PRACTICES	QAP No.	17
		Date:	10/23/02

Origination and Acceptance:

	Name	Signature	Date
Originator:	Susan Bunch	Susan Bunch	10/29/02
Quality Assurance Unit:	Susan Bunch	Susan Bunch	10/29/02
Laboratory Director:	Faust R. Parker, Jr.	Faust R. Parker, Jr.	10-29-02

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Bunch	9/25/03	Faust R. Parker, Jr.	9-26-03	No changes	1.0
Susan Bunch	9/8/05	Faust R. Parker, Jr.	9-9-05	No changes	1.0
Susan Bunch	1/29/07	Faust R. Parker, Jr.	1-29-07	No changes	1.0
Susan Bunch	10/8/08	Faust R. Parker, Jr.	10-8-08	No changes	1.0

QAP No. 17	Rev. No.: 1.0	Date: October 23, 2002

COORDINATION OF QUALITY CONTROL PRACTICES

1.0 PURPOSE

The purpose of this Quality Assurance Procedure is to describe the coordination of quality control practices at PBS&J Environmental Toxicology Laboratory.

2.0 RESPONSIBILITIES

The Quality Assurance Manager coordinates quality control practices. The Laboratory Director is responsible for the quality of all work produced. Individual SOPs state the responsibilities for individual quality control procedures.

3.0 QUALITY CONTROL IN THE LABORATORY

Quality control is maintained in the laboratory by a variety of practices. These QC practices are specific to each procedure and are stipulated in the individual SOPs. This includes test methods, equipment and instruments, chemistries, reference toxicant program, and culture practices.

4.0 QUALITY CONTROL IN DATA REVIEW

Quality control is reviewed during initial and final data review as stated in SOP 7001 and SOP 7002 and the individual test method SOPs.

5.0 INTERNAL QUALITY SYSTEMS AND OPERATIONS AUDITS

Audits performed at PBS&J serve to ensure that both operations and quality systems continue to meet set standards. Internal quality systems audits will be conducted at least once per year and operations audits will be conducted at least ten (10) times annually. The audits are explained fully in SOP1006.

6.0 NON-CONFORMANCE AND CORRECTIVE ACTION

SOP 1003 describes this procedure for detecting and documenting non-conformance issues and for implementing appropriate corrective action(s).

7.0 MANAGERIAL REVIEWS AND THE QUALITY BOARD

Managerial reviews are conducted during quarterly Quality Board meetings, through which Management (i.e., the Quality Board) has the opportunity to review and evaluate the laboratory's quality system, and testing and calibration activities. The Quality Board provides decision making to solve non-conformances and develops corrective action methods to provide quality improvement solutions. See SOP 1005 for further information.

Q A P	ETHICS AND DATA INTEGRITY	QAP No.	18
		Date:	08/22/07

Origination and Acceptance:

	Name	Signature	Date
Originator:	James D. Horne	James D Horne	8/22/07
Quality Assurance Unit:	Susan Bunch	Susan G Bunch	8/22/07
Laboratory Director:	Faust R. Parker, Jr.	Faust R Parker	8-22-07

Review and Re-Approval

Reviewer	Date	Laboratory Director	Date	Comments	Revision No.
Susan Bunch	10/8/08	Faust R Parker	10-8-08	No changes	1.0

QAP No. 18	Rev. No.: 1.0	Date: August 22, 2007

ETHICS AND DATA INTEGRITY

1.0 PURPOSE

The purpose of this Quality Assurance Procedure is to describe the PBS&J management policy and procedures for assuring and maintaining data integrity.

2.0 MANAGEMENT POLICY

The PBS&J management policy is to:

- conduct our business with integrity, honesty, decency, fairness and trustworthiness
- comply with all applicable laws, regulations, company policies and procedures, and industry best practices
- avoid conflicts of interest
- hold paramount the health and safety of the public by never sacrificing quality for profit
- embody the highest professional standards and comply with our company's values and business and professional guidelines
- prohibit retaliation of any kind against any employee who, in good faith, raises concerns or makes reports of potential misconduct.

3.0 CORE ELEMENTS AND PROCEDURES

3.1 Training

A) New hires will be given formal ethics, compliance, and data integrity training as part of their orientation. Major emphasis will be placed on the following elements:

- Corporate Ethics & Compliance Training
  - When to get assistance
  - Where to get assistance (Compliance & Ethics Hotline)
- Business Ethics Policy
- Data Fraud Training module
- Data Corrections (SOP 7004)

B) Current employees will be given an annual ethics and compliance refresher seminar; key topics will include the following elements:

- Business Ethics Policy - including data fraud module
- Recognition and reporting of data integrity issues
- Record-keeping and corrections

3.2 Monitoring

A) Original records are reviewed daily, while tests are underway, by the responsible laboratory (test-area) manager or the Laboratory Supervisor. Data inconsistencies or recording errors are resolved and corrected in accordance with SOP 7004, Data Corrections.

B) Completed data packages are reviewed by the responsible laboratory (test-area) manager or the Laboratory Supervisor in accordance with SOP 7002, Initial Data Review.

C) Final review and packaging is completed by data management personnel in accordance with SOP 7001, Final Data Review and Data Packaging. Reports are reviewed and approved, by signature, by the Laboratory Director or Technical Director, or other designee.

PBS&J is committed to reviewing or investigating–and taking appropriate action regarding–all allegations of violations of its policies and applicable laws. The review and investigation will be kept confidential to the extent possible, regardless of the outcome. PBS&J will take corrective action and make necessary changes. Anyone violating our standards of conduct will be subject to appropriate disciplinary action, up to and including termination of employment.

4.0 RECORDKEEPING

Records of initial and annual refresher training regarding ethics and data integrity issues provided to employees and agents of the PBS&J Environmental Toxicology Laboratory will be maintained by the Quality Assurance Unit.

Volume 3
STANDARD OPERATING PROCEDURES

PBS&J
Environmental Toxicology Laboratory
888 West Sam Houston Parkway South, Suite 110
Houston, TX 77042-1917
Tel: 713-977-1500
Fax: 713-977-9233

Faust R. Parker, Jr., Ph.D.
Vice President & Division Manager
Laboratory Director

James D. Horne
Technical Director

Susan Bunch
Quality Assurance Manager

Rachael Brown
Laboratory Supervisor

Matt Matthews
Client Services Manager

Standard Operating Procedures (SOPs) are designed to be reviewed and/or revised independent of each other; therefore, pagination within this Volume 3 of the Quality Assurance Manual is not sequential.

SOP #	TITLE
1001	Reference Toxicant Program
1002	Preparation of SOPs
1003	Non-conformance
1004	Vendor Approval
1005	Managerial Review and the Quality Board
1006	Audits
1007	Demonstration of Capability
1008	Health & Safety Audits
1009	Lab Technician General Training
1010	Culturist Training
1011	Management of Change
2001	Sample Check in
2002	Receipt, storage and use of standards and reagents
2003	Collection of Intermediate Samples
2004	Sample Composting
3001	Chironomus tentans Food Preparation and Feeding
3010	Artemia nauplii - Preparation for Feed
3020	Selenastrum capricornutum Culture
3030	Isochrysis galbana (marine algae) preparation & feeding
3040	YCT Preparation
3050	Culture of Branchionus plicatilis (Rotifer) for Feed
3060	Daphnia species food preparation
3070	Flake food storage and use
3080	L plumulosus Food Preparation and Feeding
3090	Evaluation of New Food used in testing and culturing
3110	Pimephales promelas Culture Practices
3120	Daphnia magna Culture Practices
3130	Dapnia pulex Culture Practices
3140	Ceriodaphnia dubia Culture Practices
3210	Menidia beryllina Culture Practices
3231	Mysidopsis bahia production system culture practices

SOP #	TITLE
3232	Mysidopsis bahia Grow-out Systems Culture Practices
3233	Mysidopsis bahia post larval culture practices
3310	Nitrobacter and Nitrosomonas Factory Culture Practices
3320	Eheim Filters in Culture
3340	Seawater preparation and maintenance of mixing tank
3350	Taxonomic Identification
4001	Static Sheen Test
4002	SDF Ammended Sediments Preparation
4003	Leptocheirus plumulosus 10d Amended Sediment
4004	Pimephales promelas embryo larva study
4005	TIE Phase I
4007	28d Closed Bottle
4008	Selanastrum capricornutum growth test (Method 1003.0)
4010	Preparation of a water accomodated fraction (WAF)
4012	PI Modified Chronic Pimephales promelas
4013	Hyalella azteca 10d Sediment Survival & Growth (ASTM E 1706)
4014	Chironomus tentans 10d Sediment Survival & Growth (ASTM E 1706)
4015	Daphnia pulex Acute WET (EPA 2021.0)
4016	Pimephales promelas Acute WET (EPA 2000.0)
4017	Mysidopsis bahia Acute WET (EPA 2007.0)
4018	Menidia beryllina Acute WET (EPA 2006.0)
4019	Cyprinodon variegatus Acute WET (EPA 2004.0)
4020	Mysidopsis bahia Chronic WET (EPA 1007.0)
4021	Pimephales promelas Chronic WET (EPA 1000.0)
4022	Ceriodaphnia dubia Chronic WET (EPA 1002.0)
4023	Menidia beryllina Chronic WET (EPA 1006.0)
4024	Cyprinodon variegatus Chronic WET (EPA 1004.0)
4025	O mykiss Acute WET (EPA 2019.0)
4026	Mysidopsis bahia 10d Sediment Survival
4027	Ampelisca abdita 10d USACE
4028	Paleomenetes pugio 10d Sed
4029	Ampelisca abdita 10d Sediment Survival (ASTM E 1367)
4030	Cyprinella leedsi Acute WET (EPA 2000.0)
4031	Ceriodaphnia dubia Acute WET (EPA 2002.0)
4032	Nereis virens 28d bioaccumulation (ASTM E 1688)

SOP #	TITLE
4033	Macoma nasuta 28d bioaccumulation (ASTM E 1688)
4034	Lumbriculus variegatus 28d bioaccumulation (EPA 100.3)
4035	Daphnia magna Chronic WET
4036	Mercenaria merceneria 28d bioaccumulation (ASTM E 1688)
4038	Reverse Phase Extraction (RPE) Test for Free Oil Contamination
4039	Mysidopsid bahia, Low Salinity Acclimation
4040	Ampelisca abdita and Americamysis bahia 7d
4041	Leptocheirus plumulosus 10d Sediment Survival (ASTM E 1367)
4042	Mercenaria mercenaria 7d WST
4043	Corbicula fluminea 28d bioaccumulation (ASTM E 1688)
4044	Hyalella azteca 10d Sediment Survival & Growth (EPA 100.1)
4045	Chironomus tentans 10d Sediment Survival & Growth (EPA 100.2)
4046	Elutriate Preparation for Water Column Bioassay
5001	Incident illuminance
5002	Measuring D.O. and calibration of meter
5003	Measuring SCT and calibration of SCT meter YSI model 30
5004	Measuring SCT and calibration of SCT meter YSI model 3100
5006	pH Meter Orion 210A
5007	pH Meter Orion 410A
5008	pH Meter Orion 3 Star
5009	Algae Density by Hemocytometer
5010	Algae Density by Spec
5012	Laboratory Thermometers Calibration & Use
5013	Ohaus Analytical balance; use and calibration
5014	Class S Weights Use and Maintenance
5015	Fluoride Measurement using Hach DR DR/3000 Spec.
5016	Ammonia probe
5017	Calibration & Operation of YSI 3256 Conductivity Cell
5020	Equipment Maintenance Scheduling
5021	Facility Maintenance-outside service
6001	Determination of total hardness
6002	Determination of total alkalinity
6003	Determination of total residual chlorine
6004	Sample Dechlorination
7001	Final data review and packaging

SOP #	TITLE
7002	Initial Data Review
7003	Electronic Document Storage
7004	Data Corrections
9001	Laboratory temperature control
9002	Acid bath system, use and maintenance
9003	Glass and plastic ware cleaning
9004	Laboratory photoperiod verification
9005	Synthetic seawater transfer and acceptability
9006	Nalgene Tank Maintenance
9007	Synthetic Freshwater Preparation
9008	Water Vessel and Eheim Maintenance
9009	Sample Storage and Disposition
9010	Verification of Reagent Grade Water Quality
9011	Sample Kit Preparation
9012	ISCO Model 3700 Portable Sampler

Appendix C-2

Columbia Analytical Services - Quality Assurance Manual

QUALITY ASSURANCE MANUAL

Columbia Analytical Services, Inc.

1317 South 13th Avenue
Kelso, Washington 98626
(360) 577-7222

Effective Date: October 30, 2009

Laboratory Director/Technical Director: _____
Jeff Christian

Quality Assurance Manager: _____
Julie Gish

Technical Director – Metals: _____
Jeff Coronado

Technical Director – Metals R & D: _____
Nicholas Bloom

Technical Director – Organics: _____
Jeff Grindstaff

Technical Director – Organic Extractions: _____
Dr. Gregory Salata

Technical Director – Inorganics/Microbiology: _____
Harvey Jacky

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

2.0 TABLE OF CONTENTS

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3.0 INTRODUCTION AND COMPANY QUALITY ASSURANCE POLICY

Columbia Analytical Services, Inc. (CAS) is an employee-owned professional analytical services laboratory which performs chemical and microbiological analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sludge, sediment, tissue, industrial and hazardous waste, and other material.

Quality Management Systems are established, implemented and maintained by management. Systems are designed so that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. Quality Systems are applicable to all fields of testing in which the laboratory in involved.

This goal is achieved by ensuring that adequate Quality Control (QC) procedures are used throughout the monitoring process, and by establishing a means to assess performance of these Quality Control and other QA activities. Policies and procedures are established in order to meet the quality objectives of clients, accrediting authorities, and certifying organizations. Columbia Analytical Services, Inc. is committed to operate in accordance to: ISO/IEC 17025:2005 International Standards, The NELAC Institute (TNI) National Environmental Laboratory Accreditation Program (NELAP), and DoD Environmental Laboratory Accreditation Program. Quality Systems are established to meet the requirements of these standards.

Laboratory management is committed to continually improve the effectiveness of its quality systems and to ensure that all tests are carried out in accordance to customer requirements. Key elements of this commitment are set fourth in the *Columbia Analytical Services, Inc. Quality and Ethics Policy Statement March* 2009 and in this *Kelso Quality Assurance Manual* (QAM). We recognize that quality assurance requires a commitment to quality by everyone in the organization - individually, within each operating unit, and throughout the entire laboratory.

Columbia Analytical maintains control of analytical results by adhering to written standard operating procedures (SOPs) and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data.

Columbia Analytical is a network of laboratories. In addition to the Kelso, WA facility, to which this manual is applicable, Columbia Analytical also operates laboratories in California, Florida, New York, Arizona, and Texas.

The information in this document has been organized according to the format described in *EPA Requirements for Quality Management Plans, EPA QA/R-2,* USEPA, 2001; *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5,* USEPA, 2001, and *ISO17025 International Standard.*

4.0 PROGRAM DESCRIPTION

The purpose of the QA program at Columbia Analytical is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality. The concept of Quality Assurance can be extended, and is expressed in the mission statement of Columbia Analytical:

> "The mission of Columbia Analytical Services, Inc. is to provide high quality, cost-effective, and timely professional testing services to our customers. We recognize that our success as a company is based on our ability to maintain customer satisfaction. To do this requires constant attention to customer needs, maintenance of state-of-the-art testing capabilities and successful management of our most important asset - our people - in a way that encourages professional growth, personal development and company commitment."

4.1 Quality Management Systems

In support of this mission, the Kelso laboratory has developed Quality Management Systems to ensure all products and services meet our client's needs. These systems incorporate the requirements of ISO17025 standards. Quality Management Systems Include:

- Standard Operating Procedures
- Sample Management
- Chain of Custody Procedures
- Statistical Control Charting
- Standards Traceability
- Core Ethics Training
- Document Control
- Corrective Action Program
- Management Reviews
- Demonstration of Capability

The effectiveness of the Quality Management System is assessed in several ways:

- Internal and External Audits covering all aspects of the organization
- Annual Management Reviews
- Analysis of Customer Complaints
- Internal and External Proficiency Testing

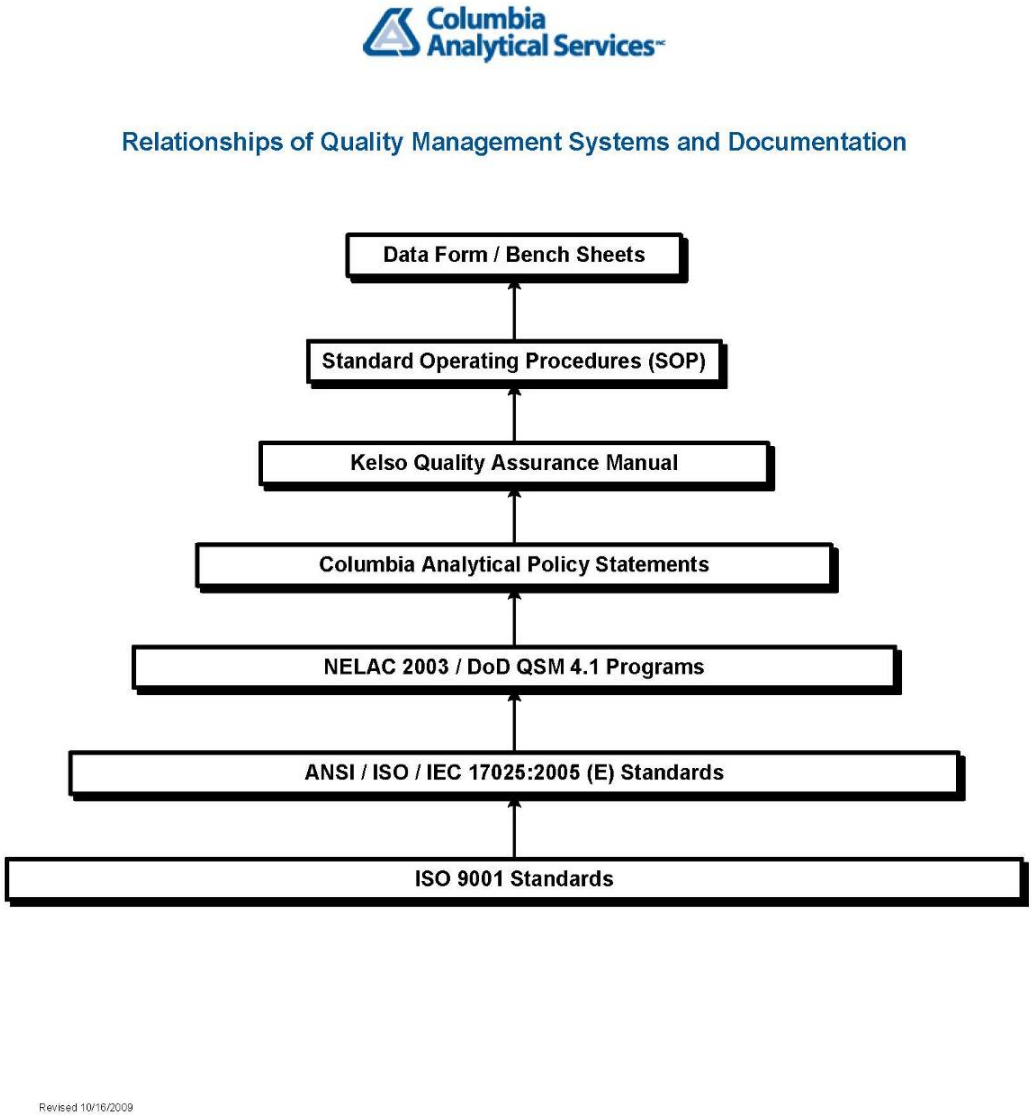


Figure 4-1

Kelso Quality Management Systems are based upon ISO 17025:2005 standards. Fundamental programs (NELAC 2003 and DoD QSM) are based upon these standards. Implementation and documentation against these standards are communicated in corporate policy statements, and Kelso's Quality Assurance Manual. Actual procedures, actions and documentation are defined in both administrative and technical SOP's.

4.2 Facilities and Equipment

Columbia Analytical features over 45,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system has been specially designed to meet the needs of the analyses performed in each work space. Also, Columbia Analytical minimizes laboratory contamination sources by employing janitorial and maintenance staff to ensure that good housekeeping and facilities maintenance are performed. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types. These specialized areas (and access restrictions) include:

- Shipping and Receiving/Purchasing
- Sample Management Office, including controlled-access sample storage areas
- Inorganic/Metals Sample Preparation Laboratories (2)
- Inorganic/Metals "clean room" sample preparation laboratory
- ICP-AES Laboratory
- ICP-MS Laboratory
- AA Laboratory
- Metals R&D Laboratory
- Water Chemistry & General Chemistry Laboratories (3)
- Semi-volatile Organics Sample Preparation Laboratory
- Gas Chromatography/High Performance Liquid Chromatography Laboratories
- Gas Chromatography/Mass Spectrometry Laboratory
- Petroleum Hydrocarbon Laboratory
- Semi-volatile Organics Drinking Water Laboratories (2)
- Volatile Organics Laboratory
    - Separate sample preparation laboratory
    - Access by semi-volatile sample preparation staff only after removing lab coat and solvent-contaminated gloves, etc.
- Microbiology Laboratory
- Laboratory Deionized Water Systems (2)
- Laboratory Management, Client Service, Report Generation and Administration
- Data Archival, Data Review and support functions areas
- Information Technology (IT) and LIMS

In addition, the designated areas for sample receiving, refrigerated sample storage, dedicated sample container preparation and shipping provide for the efficient and safe handling of a variety of sample types. Figure 4-1 shows the facility floor plan. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation are appropriate for the procedures in use. Appendix C lists the major equipment, illustrating the laboratory's overall capabilities and depth.

4.3 Technical Elements of the Quality Assurance Program

The laboratory's technical procedures are based upon procedures published by various agencies or organizations (See Section 18). The Quality Assurance Program provides to the laboratory organization, procedures, and policies by which the laboratory operates. The necessary certifications and approvals administered by external agencies are maintained by the QA department. This includes method approvals and audit administration. In addition,

internal audits are performed to assess compliance with policies and procedures. Standard Operating Procedures (SOPs) are maintained for technical and administrative functions. A document control system is used for SOPs, as well as laboratory notebooks, and this QA Manual. A list of QA Program documents is provided in Appendix A.

Acceptable calibration procedures are defined in the SOP for each test procedure. Calibration procedures for other laboratory equipment (balances, thermometers, etc.) are also defined. Quality Control (QC) procedures are used to monitor the testing performed. Each analytical procedure has associated QC requirements to be achieved in order to demonstrate data quality. The use of method detection limit studies, control charting, technical training and preventative maintenance procedures further ensure the quality of data produced. Proficiency Testing (PT) samples are used as an external means of monitoring the quality and proficiency of the laboratory. PT samples are obtained from qualified vendors and are performed on a regular basis. In addition to method proficiency, documentation of analyst training is performed to ensure proficiency and competency of laboratory analysts and technicians. Sample handling and custody procedures are defined in SOPs. Procedures are also in place to monitor the sample storage areas. The technical elements of the QA program are discussed in further detail in later sections of this QA manual.

4.4 Operational Assessments

The laboratory uses a number of systems to assess its daily operations. In addition to the routine quality control (QC) measurements, the senior laboratory management examines a number of other indicators to assess the overall ability of the laboratory to successfully perform analyses for its clients including; On-time performance, customer complaints, training reports and non-conformity reports. A frequent, routine assessment must also be made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload.

Columbia Analytical utilizes a number of different methods to ensure that adequate resources are available in anticipation of the demand for service. Regularly scheduled senior staff meetings, tracking of outstanding proposals and an accurate, current synopsis of incoming work all assist the senior staff in properly allocating resources to achieve the required results. All Requests for Proposal (RFP) documents are reviewed by the Project Chemist and appropriate managerial staff to identify any project specific requirements that differ from the standard practices of the laboratory. Any requirements that cannot be met are noted and communicated to the client, as well as requesting the client to provide any project specific Quality Assurance Plans (QAPPs) if available. A weekly status meeting is also conducted with the laboratory staff by the Client Services Manager to inform the staff of the status of incoming work, future projects, or project requirements.

4.5 Document Control

Procedures for control and maintenance of documents are described in the *SOP for Document Control (ADM-DOC_CTRL).* The requirements of the SOP apply to all standards preparation logbooks, instrument maintenance logbooks, run logbooks, certificates of analysis, standard operating procedures (SOPs), quality assurance manuals (QAMs), quality assurance project plans (QAPPs), Environmental Health & Safety (EHS) manuals, and other controlled Columbia Analytical documents.

Each controlled copy of a controlled document will be released only after a document control number is assigned and the recipient is recorded on a document distribution list. Filing and distribution is performed by the Quality Assurance Manager, or designee, and ensure that only the most current version of the document is distributed and in use. A document control number is assigned to logbooks. Completed logbooks that are no longer in use are archived in a master logbook file.

Columbia Analytical maintains a records system that ensures all laboratory records (including raw data, reports, and supporting records) are retained and available. The archiving system is described in the *SOP for Data Archiving (ADM-ARCH).*

4.6 Subcontracting

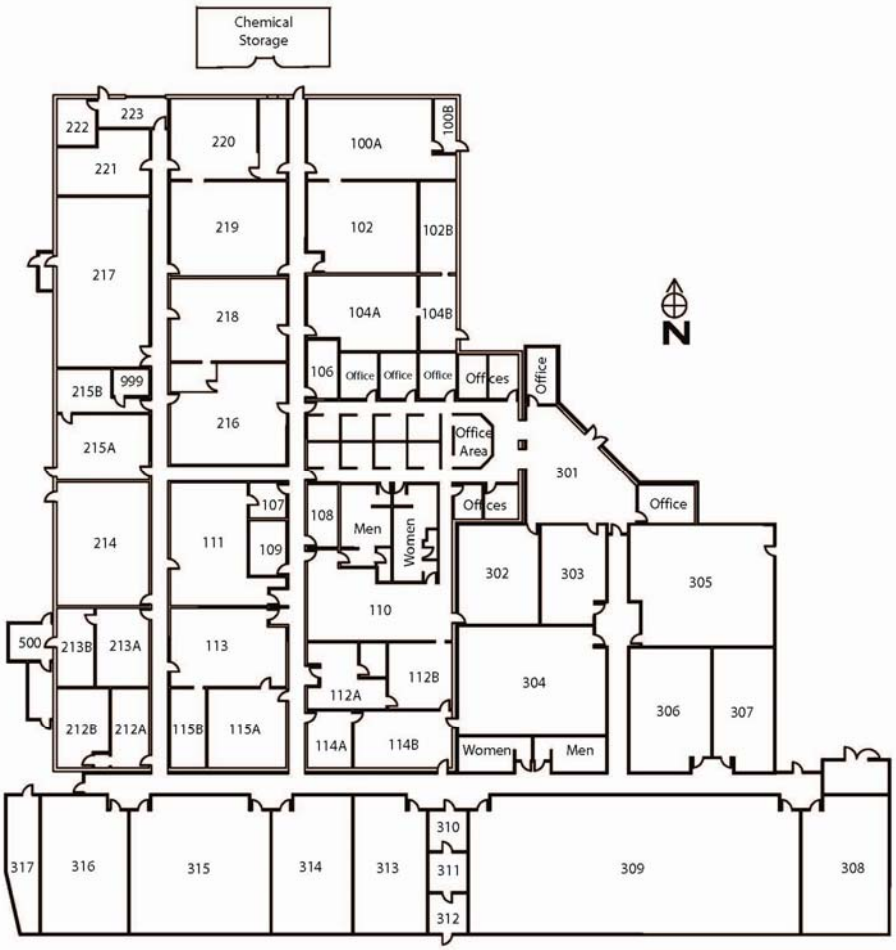
Analytical services are subcontracted when Columbia Analytical/Kelso needs to balance workload or when the requested analyses are not performed by Columbia Analytical/Kelso. Subcontracting is only done with the knowledge and approval of the client and to qualified laboratories. Subcontracting to another Columbia Analytical laboratory is preferred over external-laboratory subcontracting. Further, sub-contracting is done using capable and qualified laboratories. Established procedures are used to qualify external subcontract laboratories. These procedures are described in the *SOP for Qualification of Subcontract Laboratories (ADM-SUBLAB).* The Corporate Quality Assurance staff is responsible for qualifying and oversight of subcontract laboratories.

4.7 Procurement

The quality level of reagents and materials (grade, traceability, etc.) required is specified in analytical SOPs. Department supervisors ensure that the proper materials are purchased. Inspection and verification of material ordered is performed at the time of receipt by receiving personnel. The receiving staff labels the material with the date received. Expiration dates are assigned (by the laboratory user) as appropriate for the material. Storage conditions and expiration dates are specified in the analytical SOP. Supplies and services that are critical in maintaining the quality of laboratory testing are procured from pre-approved vendors. The policy and procedure for purchasing and procurement are described in the *SOP for Purchasing through CAS Purchasing Department in Kelso (SOP ADM-PUR).* Also, refer to section 10.4 for a discussion of reference materials.

Figure 4-2
Columbia Analytical/Kelso Laboratory Floor Plan

Room #	
100A	Semivolatile GC Lab
100B	Electrical Room
102	Semivolatile GC Lab
102B	LC Lab
104A	Petroleum Hydrocarbon Lab
104B	Petroleum Hydrocarbon Lab
106	Deionized Water System Room
107	Storage
108	Zero Headspace Extraction
109	Storage
110	Lunch Room
111	General Chemistry Lab
112A	Microbiology Lab
112B	Copy Center
113	Metals Lab
114A	Sample Preparation
114B	Sample Preparation
115A	Metals Lab
115B	Metals Lab
212A	Metals Lab
212B	Low Level Mercury Lab
213A	Sample Preparation
213B	Sample Preparation
214	General Chemistry Lab
215A	Drinking Water Lab
215B	IT
216	Data Review Area
217	Sample Storage
218	Volatiles Lab
219	Semivolatile GC/MS Lab
220	Semivolatile GC/MS Lab
221	Drinking Water Lab
222	Purchasing
223	General Receiving
301	Lobby
302	Lunch Room
303	Conference Room
304	Sample Storage
305	Sample Receiving
306	Metals R & D
307	Pharmaceutical B
308	Pharmaceutical A
309	Organics Extractions Lab
310	Communication Room
311	Electrical Room
312	Fire Room/Water System
313	Glass Room
314	Clean Room
315	Metals Digestion Room
316	Tissue Preparation
317	Mechanical
500	Janitorial Room
999	Computer Room



5.0 PROFESSIONAL CONDUCT AND ETHICAL PRACTICES

One of the most important aspects of the success of Columbia Analytical is the emphasis placed on the integrity of the data provided and services performed. To promote product quality, employees are required to comply with certain standards of conduct and ethical practices. The following examples of Columbia Analytical policy are representative of these standards, and are not intended to be limiting or all-inclusive:

- Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action. Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.
- Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible. Employee discipline is progressive in its severity and each situation is handled individually in that the discipline is designed to fit the circumstances. Potential disciplinary actions may include a verbal warning, written warning, a second written notice (more severe and more strongly worded than a warning), suspension without pay, demotion, or termination.
- It is the responsibility of all Columbia Analytical employees to safeguard sensitive company and client information. The nature of our business and the well being of our company and of our clients is dependent upon protecting and maintaining proprietary company/client information. All information, data, and reports (except that in the public domain) collected or assembled on behalf of a client is treated as confidential. Information may not be given to third parties without the consent of the client. Unauthorized release of confidential information about the company or its clients is taken seriously and is subject to formal disciplinary action.

All employees are required to sign and adhere to the requirements set forth in the Columbia Analytical *Confidentiality and Conflicts of Interest Employee Agreement* and the Columbia Analytical *Commitment to Excellence in Data Quality Policy.* All employees receive in-house ethics training and are periodically reminded of their data quality and ethical conduct responsibilities.

Columbia Analytical makes every attempt to ensure that employees are free from any commercial, financial, or other undue pressures that might affect their quality of work. Related policies are described in the Columbia Analytical Employee Handbook. This includes the Columbia Analytical Ombudsman Program, the Columbia Analytical Open Door Policy, and the use of flexible work hours. Operational assessments are regularly made to ensure that project planning is performed and that adequate resources are available during anticipated periods of increased workloads (Section 4.3). Procedures for subcontracting work are established, and within the Columbia Analytical laboratory network additional capacity is typically available for subcontracting, if necessary.

6.0 ORGANIZATION AND RESPONSIBILITIES

The Columbia Analytical/Kelso staff, consisting of approximately 130 employees, includes chemists, technicians and support personnel. They represent diverse educational backgrounds and experience, and provide the comprehensive skills that the laboratory requires. During seasonal workload increases, additional temporary employees may be hired to perform specific tasks.

Columbia Analytical is committed to providing an environment that encourages excellence. Everyone within Columbia Analytical shares responsibility for maintaining and improving the quality of our analytical services. The responsibilities of key personnel within the laboratory are described below. Table 6-1 lists the Columbia Analytical/Kelso personnel assigned to these key positions. Managerial staff members are provided the authority and resources needed to perform their duties. An organizational chart of the laboratory, as well as the resumes of these key personnel, can be found in Appendix B.

- The role of the **Laboratory Director** is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program and is responsible for overall laboratory efficiency and the financial performance of the Kelso facility. The Laboratory Director has the authority to stop work in response to quality problems. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.

- The responsibility of the **Quality Assurance Manager** (QAM) is to oversee implementation of the quality program and to coordinate QA activities within the laboratory. The QAM works with laboratory production units to establish effective quality control and assessment plans. The QAM has the authority to stop work in response to quality problems. The QAM is responsible for maintaining the QA Manual and performing an annual review of it; reviewing and approving SOPs and coordinating the annual review of each SOP; maintaining QA records such as metrological records, archived logbooks, PT sample results, etc.; document control; conducting PT sample studies; approving nonconformity and corrective action reports; maintaining the laboratory's certifications and approvals; performing internal QA audits; preparing QA activity reports; etc. The QAM reports directly to the Laboratory Director. The QAM also interacts with the Columbia Analytical Quality Assurance Director. It is important to note that when evaluating data, the QAM does so in an objective manner and free of outside, or managerial, influence.

  The Chief Quality Officer (CQO) is responsible for the overall QA program at all the Columbia Analytical laboratories. The CQO is responsible for ensuring that annual internal audits are performed at each Columbia Analytical laboratory; maintaining a data base of information about state certifications and accreditation programs; writing laboratory-wide SOPs; maintaining a data base of Columbia Analytical-approved subcontract laboratories; providing assistance to the laboratory QA staff and laboratory managers; preparing a quarterly QA activity report; etc.

- In the case of absence of the Laboratory Director or QA Manager, deputies are assigned to act in that role. Default deputies for these positions are the Client Services Manager or Organics Department Manager (for the Laboratory Director) and the CQO or Laboratory Director (for the QA Manager).

- The **Environmental Health and Safety Officer** (EH&S) is responsible for the administration of the laboratory health and safety policies. This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections. The EH&S officer is also designated as the Chemical Hygiene Officer. The EH&S Officer has a dotted-line reporting responsibility to Columbia Analytical's EH&S Director.

- The **Client Services and Sample Management Office Manager** is responsible for the Client Services Department (customer services/project chemists, and Electronic Data Deliverables group) and the sample management office/bottle preparation sections. The Client Services Department provides a complete interface with clients from initial project specification to final deliverables. The sample management office handles all the activities associated with receiving, storage, and disposal of samples. The Client Services Manager has the authority to stop subcontractor work in response to quality problems.

- The **Project Chemist** is a senior-level scientist assigned to each client to act as a technical liaison between the client and the laboratory. The project chemist is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements. This entails coordinating with the Columbia Analytical laboratory and administrative staff to ensure that client-specific needs are understood, and that the services Columbia Analytical provides are properly executed and satisfy the requirements of the client.

- The Analytical Laboratory is divided into operational units based upon specific disciplines. Each department is responsible for establishing, maintaining and documenting a quality control program based upon the unique requirements within the department. Each **Department Manager and Supervisor** has the responsibility to ensure that quality control functions are carried out as planned, and to guarantee the production of high quality data. Department managers and bench-level supervisors have the responsibility to monitor the day-to-day operations to ensure that productivity and data quality objectives are met. Each department manager has the authority to stop work in response to quality problems in their area. Analysts have the responsibility to carry out testing according to prescribed methods, SOPs, and quality control guidelines particular to the laboratory in which he/she is working.

- The **Sample Management Office** plays a key role in the laboratory QA program by maintaining documentation for all samples received by the laboratory, and by assisting in the archival of all laboratory results. The sample management office staff is also responsible for the proper disposal of samples after analysis.

- **Information Technology** (IT) staff are responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, IT systems development and implementation, education of analytical staff in the use of scientific software, Electronic Data Deliverable (EDD) generation, and data back-up, archival and integrity operations.

Table 6-1
Summary of Technical Experience and Qualifications

Personnel	Years of Experience	Project Role
Jeff Christian, B.S.	30	Laboratory Director
Julie Gish, M.S.	18	Quality Assurance Manager
Lynda Huckestein, B.S.	20	Client Services Manager Sample Management Office Manager
Jeff Coronado, B.S.	19	Metals Department Manager
Nicolas Bloom, M. S.	29	Metals R & D Manager
Harvey Jacky, B.S.	20	General Chemistry Department Manager
Gregory Salata, Ph.D.	9	Extractions Department Manager
Jeff Grindstaff, B.S.	20	Organics Chromatography & Mass Spectrometry Department Manager
Loren Portwood, B.S.	18	Organics Drinking Water Department Manager
Eileen Arnold, B.A.	27	Environmental Health and Safety Officer
Mike Sullivan, B.S.	8	Information Technology Director
Lee Wolf, B.S.	23	Chief Quality Officer
Steve Vincent, B.S.	33	President

7.0 INFORMATION MANAGEMENT

The generation, compilation, reporting, and archiving of electronic data is a critical component of laboratory operations. In order to generate data of known and acceptable quality, the quality assurance systems and quality control practices for electronic data systems must be complete and comprehensive and in keeping with the overall quality assurance objectives of the organization. Columbia Analytical management provides the tools and resources to implement electronic data systems and establishes information technology standards and policies. Appendix C lists major automated data processing equipment.

7.1 Software Quality Assurance Plan

Columbia Analytical has defined practices for assuring the quality of the computer software used throughout all laboratory operations to generate, compile, report, and store electronic data. These practices are described in the *CAS Software Quality Assurance Plan (SQAP)*. The purpose of the SQAP is to describe the policies and practices for the procurement, configuration management, development, validation and verification, data security, maintenance, and use of computer software. The policies and practices described in the plan apply to purchased computer software as well as to internally developed computer software. Key components of this plan are policies for software validation and control.

7.2 IT Support

The local Columbia Analytical Information Technology (IT) department is established to provide technical support for all computing systems. The IT department staff continually monitors the performance and output of operating systems. The IT department oversees routine system maintenance and data backups to ensure the integrity of all electronic data. A software inventory is maintained. Additional IT responsibilities are described in the SQAP.

In addition to the local IT department, Columbia Analytical corporate IT provides support for network-wide systems. Columbia Analytical also has personnel assigned to information management duties such as development and implementation of reporting systems; data acquisition, and Electronic Data Deliverable (EDD) generation.

7.3 Information Management Systems

Columbia Analytical has various systems in place to address specific data management needs. The Columbia Analytical Laboratory Information Management System (LIMS) is used to manage sample information and invoicing. Access is controlled by password. This system defines sample identification, analysis specifications, and provides a means of sample tracking. This system is used during sample login to generate the internal service request. Included on the service request is a summary of client information, sample identification, required analyses, work instructions, deliverable requirements. The LIMS is used to track the status of a sample and is important in maintaining internal chain of custody.

Where possible, instrument data acquired locally is immediately moved to a server (Microsoft Windows2003® domain). This provides a reliable, easily maintained, high-volume acquisition and storage system for electronic data files. With password entry, users may access the system from many available computer stations, improving efficiency and flexibility. The server is also used for data reporting, EDD generation, and administrative functions. Access to these systems is controlled by password. A standardized EDI (electronic data interchange) format is used as a reporting platform, providing functionality and flexibility for end users. With a common standardized communication platform, the EDI provides data reporting in a variety of hardcopy and electronic deliverable formats, including Staged Electronic Data Deliverable (SEDD) format.

7.4 Backup and Security

Columbia Analytical laboratory data is either acquired directly to the centralized acquisition server or acquired locally and then transferred to the server. All data is eventually moved to the centralized data acquisition server for reporting and archiving. Differential backups are performed on all file server information once per day, Sunday through Thursday. Full backups are performed each Friday night. Tapes are physically stored in a locked media cabinet within a locked, temperature controlled computer room, with every other full backup also securely stored offsite.

Access to sample information and data is on a need-to-know basis. Access is restricted to the person's areas of responsibility. Passwords are required on all systems. No direct external, non- Columbia Analytical access is allowed to any of our network systems.

The external e-mail system and Internet access is established via a single gateway to discourage unauthorized entry. Columbia Analytical uses a closed system for company e-mail. Files, such as electronic deliverables, are sent through the external e-mail system only via a trusted agent. The external messaging system operates through a single secure gateway. Email attachments sent in and out of the gateway are subject to a virus scan. Because the Internet is not regulated, we use a limited access approach to provide a firewall for added security. Virus screening is performed continuously on all network systems.

8.0 SAMPLE MANAGEMENT

8.1 Sampling and Sample Preservation

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples. Columbia Analytical recommends that clients follow sampling guidelines described in 40 CFR 136, 40 CFR 141, USEPA SW-846, and state-specific sampling guidelines, if applicable. Sampling factors that must be taken into account to insure accurate, defensible analytical results include:

- Amount of sample taken
- Type of container used
- Type of sample preservation
- Sample storage time
- Proper custodial documentation

Columbia Analytical uses the sample preservation, container, and holding-time recommendations published in a number of documents. The primary documents of reference are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IV for hazardous waste samples; USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; EPA 40CFR parts 136 and 141; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples (see Section 18 for complete citations). The container, preservation and holding time information for these references is summarized in Table 8-1 for soil, water, and drinking water. The current EPA CLP Statement of Work should be referred to for CLP procedures. Where allowed by project sampling and analysis protocols (such as Puget Sound Protocols) the holding time for sediment, soil, and tissue samples may be extended for a defined period when stored frozen at -20°C.

Columbia Analytical routinely provides sample containers with appropriate preservatives for our clients. Containers are purchased as precleaned to a level 1 status, and conform to the requirements for samples established by the USEPA. Certificates of analysis for the sample containers are available to clients if requested. Reagent water used for sampling blanks (trip blanks, etc.) and chemical preservation reagents are tested by the laboratory to ensure that they are free of interferences and documented. Our sample kits typically consist of foam-lined, precleaned shipping coolers, (cleaned inside and out with appropriate cleaner, rinsed thoroughly and air-dried), specially prepared and labeled sample containers individually wrapped in protective material, (VOC vials are placed in a specially made, foam holder), chain-of-custody (COC) forms, and custody seals. Container labels and custody seals are provided for each container.

Figure 8-1 shows the chain-of-custody form routinely used at Columbia Analytical and included with sample kits. For large sample container shipments, the containers may be shipped in their original boxes. Such shipments will consist of several boxes of labeled sample containers and sufficient materials (bubble wrap, COC forms, custody seals, shipping coolers, etc.) to allow the sampling personnel to process the sample containers and return them to Columbia Analytical. The proper preservative is added to the sample containers prior to shipment, unless otherwise instructed by the client.

If any returning shipping cooler exhibits an odor or other abnormality after receipt and subsequent decontamination by laboratory personnel, a second, more vigorous decontamination process is employed. Containers exhibiting an odor or abnormality after the second decontamination process are promptly and properly discarded. Columbia Analytical keeps client-specific shipping requirements on file and utilizes major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. Columbia Analytical also provides courier service that makes regularly scheduled trips to the Greater Portland, Oregon Metropolitan area.

When Columbia Analytical ships environmental samples to other laboratories for analysis each sample bottle is wrapped in protective material and placed in a plastic bag (preferably Ziploc®) to avoid any possible cross-contamination of samples during shipping. The sample management office (SMO) follows formalized procedures (SMO-GEN) for maintaining the samples' chain of custody, packaging and shipment. Dry ice gel ice is the only temperature preservative used by Columbia Analytical, unless otherwise specified by the client or receiving laboratory.

8.2 Sample Receipt and Handling

Standard Operating Procedures (SMO-GEN) are established for the receiving of samples into the laboratory. These procedures ensure that samples are received and properly logged into the laboratory, and that all associated documentation, including chain of custody forms, is complete and consistent with the samples received.

Once samples are delivered to the Columbia Analytical sample management office (SMO), a Cooler Receipt and Preservation Check Form (CRF - See Figure 8-2 for an example) is used to assess the shipping cooler and its contents as received by the laboratory personnel. Verification of sample integrity includes the following activities:

- Assessment of custody seal presence/absence, location and signature;
- Temperature of sample containers upon receipt;
- Chain of custody documents properly used (entries in ink, signature present, etc.);
- Sample containers checked for integrity (broken, leaking, etc.);

- Sample is clearly marked and dated (bottle labels complete with required information);
- Appropriate containers (size, type) are received for the requested analyses;
- The minimum amount of sample material is provided for the analysis.
- Sample container labels and/or tags agree with chain of custody entries (identification, required analyses, etc.);
- Assessment of proper sample preservation (if inadequate, corrective action is employed); and
- VOC containers are inspected for the presence/absence of bubbles. (Assessment of proper preservation of VOC containers is performed by lab personnel).

Samples are logged into a Laboratory Information Management System (LIMS). Any anomalies or discrepancies observed during the initial assessment are recorded on the CRF and COC documents. Potential problems with a sample shipment are addressed by contacting the client and discussing the pertinent issues. When the Project Chemist and client have reached a satisfactory resolution, the login process may continue and analysis may begin. During the login process, each sample is given a unique laboratory code and a service request form is generated. The LIMS generates a Service Request that contains client information, sample descriptions, sample matrix information, required analyses, sample collection dates, analysis due dates and other pertinent information. The service request is reviewed by the appropriate Project Chemist for accuracy, completeness, and consistency of requested analyses and for client project objectives.

Samples are stored as per method requirements until they undergo analysis, unless otherwise specified, using various refrigerators or freezers, or designated secure areas. Columbia Analytical has five walk-in cold storage units which house the majority of sample containers received at the laboratory. In addition, there are four additional refrigerators, including dedicated refrigerated storage of VOC samples. The dedicated storage areas for VOC samples are monitored using storage blanks, as described in the *SOP for VOA Storage Blanks (VOC-BLAN)*. Columbia Analytical also has seven sub-zero freezers capable of storing samples at -20° C primarily used for tissue and sediment samples requiring specialized storage conditions. The temperature of each sample storage unit is monitored daily and the data recorded in a bound logbook. Continuous-graph temperature recorders have also been placed in the walk-in refrigerators to provide a permanent record of the storage conditions to which samples are exposed.

Columbia Analytical adheres to the method-prescribed or project-specified holding times for all analyses. The sampling date and time are entered into the LIMS system at the time of sample receipt and login. Analysts then monitor holding times by obtaining analysis-specific reports from the LIMS. These reports provide holding time information on all samples for the analysis, calculated from the sampling date and the holding time requirement. To document holding time compliance, the date and time analyzed is printed or written on the analytical raw data. For analyses with a holding time prescribed in hours it is essential that the sample collection time is provided, so holding time compliance can be demonstrated. If not, the sample collection time is assumed as the earliest in the day (i.e. the most conservative).

Unless other arrangements have been made in advance, upon completion of all analyses and submittal of the final report, aqueous samples and sample extracts are retained at ambient temperature for 30 days, soil samples are retained at ambient temperature for 60 days, and tissue samples are retained frozen for 3 months. Upon expiration of these time limits, the samples are either returned to the client or disposed of according to approved disposal practices. All samples are characterized according to hazardous/non-hazardous waste criteria and are segregated accordingly. All hazardous waste samples are disposed of according to formal procedures outlined in the *CAS Environmental Health and Safety Manual.* All waste produced at the laboratory, including the laboratory's own various hazardous waste streams, is treated in accordance with applicable local and Federal laws. Documentation is maintained for each sample from initial receipt through final disposal to ensure that an accurate history of the sample from "cradle to grave" is available.

8.3 Sample Custody

Sample custody transfer at the time of sample receipt is documented using chain-of-custody (COC) forms accompanying the samples. During sample receipt, it is also noted if custody seals were present. This is described in the *SOP for Sample Receiving (SMO-GEN)*. Figure 8-1 is a copy of the chain-of-custody form routinely used at Columbia Analytical.

Facility security and access is important in maintaining the integrity of samples received at Columbia Analytical/Kelso. Access to the laboratory facility is limited by use of locked exterior doors with a coded entry, except for the reception area and sample receiving doors, which are manned during business hours and locked at all other times. In addition, the sample storage area within the laboratory is a controlled access area with locked doors with a coded entry. The Columbia Analytical facility is equipped with an alarm system and Columbia Analytical employs a private security firm to provide nighttime and weekend security.

A barcoding system is used to document internal sample custody. Each person removing or returning samples from/to sample storage while performing analysis is required to document this custody transfer. The system uniquely identifies the sample container and provides an electronic record of the custody of each sample. For sample extracts and digestates the analyst documents custody of the sample extract or digestate by signing on the benchsheet, or custody record, that they have accepted custody. The procedures are described in the *SOP for Sample Tracking and Internal Chain of Custody (SMO-SCOC).*

8.4 Project Setup

The analytical method(s) used for sample analysis are chosen based on the client's requirements. Unless specified otherwise, the most recent versions of reference methods are used. For SW-846 methods, some projects may require the most recent *promulgated* version, and some projects may require the most recent *published* version. The Project Chemist will ensure that the correct method version is used. LIMS codes are chosen to identify the analysis method used for analysis. The Project Chemist ensures that the correct methods are selected for analysis, deliverable requirements are identified, and due dates are specified on the service request. To communicate and specify project-specific requirements, a Tier V form (Figure 8-3) is used and accompanies the service request form.

Table 8-1
Sample Preservation and Holding Times

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Bacterial Tests				
Coliform, Colilert (Standard Methods)	W, DW	P, Bottle or Bag	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Coliform, Fecal and Total (Standard Methods)	W, DW	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Fecal Streptococci (SM 9230B)	W	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Inorganic Tests				
Acidity (SM 2310B)	W	P,G	Cool, 4°C	14 days[EPA]
Alkalinity (SM 2320B)	W, DW	P,G	Cool, 4°C	14 days[EPA]
Ammonia (SM 4500NH3)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Biochemical Oxygen Demand (SM 5210B)	W	P,G	Cool, 4°C	48 hours
Bromate (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	28 days
Bromide (EPA 300.1)	W, DW	P,G	None Required	28 days
Chemical Oxygen Demand (SM 5220C)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Chloride (EPA 300.0)	W, DW	P,G	None Required	28 days
Chloride (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Chlorine, Total Residual (SM 4500Cl F)	W, DW	P,G	None Required	24 hours
Chlorite (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	14 days
Chlorophyll-A (SM 11200H)	W	G Amber	Cool, 4°C	Analyze immediately
Chromium VI (EPA 7196A)	W	P,G	Cool, 4°C	24 hours
Color (SM 2120B)	W, DW	P,G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination (EPA 335.4, 9010, 9012) (SM 4500CN E,G)	W, DW	P,G	Cool, 4°C, NaOH to pH>12, plus 0.6 g Ascorbic Acid	14 days
Cyanide, Weak Acid Dissociable (SM 4500CN I)	W	P,G	Cool, 4°C, NaOH to pH >12	14 days
Ferrous Iron (CAS SOP)	W, DW	G Amber	Cool, 4°C	24 hours
Fluoride (EPA 300.0)	W, DW	P,G	None Required	28 days
Fluoride (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Hardness (SM 2340C)	W, DW	P,G	HNO_3 to pH<2	6 months
Hydrogen Ion (pH) (SM 4500H B)	W, DW	P,G	None Required	Analyze immediately
Kjeldahl and Organic Nitrogen (ASTM D3590-89)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Nitrate (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrate (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrate (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Nitrate-Nitrite (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrite (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrite (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrite (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Orthophosphate (EPA 365.3)	W, DW	P,G	Cool, 4°C	Analyze immediately
Oxygen, Dissolved (Probe) (SM 4500O G)	W, DW	G, Bottle and Top	None Required	Analyze immediately
Oxygen, Dissolved (Winkler)	W, DW	G, Bottle and Top	Fix on Site and Store in Dark	8 hours
Perchlorate (EPA 314.0)	W, DW	P,G	Protect from temp. extremes	28 days
Phenolics, Total (EPA 420.1)	W	G Only	Cool, 4°C, H_2SO_4 to pH<2	28 days
Phosphorus, Total (EPA 365.3)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Residue, Total (EPA 160.3 & SM 2540B)	W	P,G	Cool, 4°C	7 days
Residue, Filterable (TDS) (SM 2540C)	W	P,G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS) (SM 2540D)	W	P,G	Cool, 4°C	7 days
Residue, Settleable (SM 2540F)	W	P,G	Cool, 4°C	48 hours
Residue, Volatile (EPA 160.4)	W	P,G	Cool, 4°C	7 days
Silica (SM 4500SiO2 C)	W	P Only	Cool, 4°C	28 days
Specific Conductance (EPA 120.1 & SM 2510B)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 300.0)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Sulfide (SM 4500S2 F)	W	P,G	Cool, 4°C, Add Zinc Acetate plus Sodium Hydroxide to pH>9	7 days
Sulfite (SM 4500SO3 B)	W	P,G	None Required	24 hours
Surfactants (MBAS) (SM 5540C)	W	P,G	Cool, 4°C	48 hours
Tannin and Lignin (SM 5550B)	W	P,G	Cool, 4°C	28 days
Turbidity (EPA 180.1)	W, DW	P,G	Cool, 4°C	48 hours

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Metals				
Metals, except CrVI and Mercury	W, DW	P,G	HNO_3 to pH<2	6 months
(EPA 200.7, 200.8, 200.9, 6010, 6020)	S	G, Teflon-Lined Cap	Cool, 4°C	6 months
Chromium VI (EPA 7195/7191)	W	P,G	Cool, 4°C	24 hours
Mercury	W	P,G	HNO_3 to pH<2	28 days
(EPA 245.1, 7470, 7471)	S	P,G	Cool, 4°C	28 days
1631E	W	F	Cool, 4°C, HCl or H_2SO_4 to pH<2	90 days
1631E	S	F	Freeze < -15ºC	1 Yr
Methyl Mercury 1630	W	F	HCL to pH<2	6 months
Organic Tests				
Oil and Grease, Hexane Extractable Material (EPA 1664)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Carbon, Total (EPA 415.1, 9060 & SM 5310C)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Halogens, Total (EPA 9020)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2, No headspace	28 days
Organic Halogens, Adsorbable (EPA 1650B)	W	G, Teflon-Lined Cap	Cool, 4°C, HNO_3 to pH<2	6 months
Petroleum Hydrocarbons, Total (EPA 8015)	W	G, Teflon-Lined Cap	Cool, 4°C, HCl or H_2SO_4 to pH<2	7 days until extraction; 40 days after extraction
	S	G, Teflon-Lined Cap	Cool, 4°C	14 days until extraction; 40 days after extraction
Pharma Personal Care Products 1694	W	Amber G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2	14 days until extraction; 40 days after extraction
Nitroaromatics and Nitramines 8330, 8330B	W,S	G, Teflon-Lined Cap	Cool, 4°C	S 14, W 7 days until extraction; 40 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Organic Test				
Methanol in Process Liquid NCASI 94.03	L	G, Teflon-Lined Cap	Cool, 4°C	30 days
HAPS – Condensates NCASI 99.01		G, Teflon-Lined Cap	Cool, 4°C	14/30 days
HAPS – Impinger/Canisters NCASI 99.02			Cool, 4°C	21 days
Perfluorinated Compounds HPLC/MS/MS	W	P	Cool, 4°C	14 days until extraction; 40 days after extraction
PBDE/PBB – ROHS GC/MS			RT	40 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Volatile Organics				
Petroleum Hydrocarbons, Volatile (Gasoline-Range Organics) (EPA 8015)	W	G, Teflon-Lined Septum Cap	Cool, 4°C, HCl to pH<2 No Headspace	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C Minimize Headspace	14 days
Purgeable Halocarbons (EPA 624, 8021, 8260)	W	G, Teflon-Lined Septum Cap, No Headspace	**No Residual Chlorine Present**: HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH<2, Cool, 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE) (EPA 624, 8021, 8260)	W	G, Teflon-Lined Septum Cap, No Headspace	**No Residual Chlorine Present**: HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH<2, Cool 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.
Acrolein, Acrylonitrile, Acetonitrile (EPA 624, 8260)	W	G, Teflon-Lined Septum Cap	Adjust pH to 4-5, Cool, 4°C, No Headspace	14 days
EDB and DBCP (EPA 8260)	W,S	G, Teflon-Lined Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	28 days

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Semivolatile Organics				
Petroleum Hydrocarbons, Extractable (Diesel-Range Organics) (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Alcohols and Glycols (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Acid Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Base/Neutral Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Polynuclear Aromatic Hydrocarbons (EPA 625, 8270, 8310)	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Organochlorine Pesticides and PCBs (EPA 608, 8081, GC/MS/MS)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Organophosphorus Pesticides (EPA 8141, GC/MS/MS)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Nitrogen- and Phosphorus-Containing Pesticides (EPA 8141)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Herbicides (EPA 8151)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Organotins (CAS SOP)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Chlorinated Phenolics (EPA 1653A)	W	G, Teflon-Lined Cap	H_2SO_4 to pH<2, Cool, 4°C[g]	30 days until extraction; 30 days after extraction
Resin and Fatty Acids (NCASI 85.02)	W	G, Teflon-Lined Cap	NaOH to pH $\geq$10, Cool, 4°C[g]	30 days until extraction; 30 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Drinking Water Organics				
Purgeable Organics (EPA 524.2)	DW	G, Teflon-Lined Septum Cap	Ascorbic Acid, HCl to pH≤2, Cool, 4°C, No Headspace	14 days
EDB, DBCP, and TCP (EPA 504.1)	DW	G, Teflon-Lined Septum Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	14 days
Carbamates, Carbamoyloximes (EPA 531.1)	DW	G, Amber, Teflon-Lined Cap	1.8 mL monochloroacetic acid to pH<3; 80 mg/L $Na_2S_2O_3$ if Res.Cl.; Cool, 4°C	28 days
Chlorinated Herbicides (EPA 515.4)	DW	G, Amber, Teflon-Lined Cap	If Res.Cl, 2mg/4omL NaS; Cool, <6°C	14 days until extraction; 21 days after extraction
Chlorinated Pesticides (EPA 508.1, 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH≤ 2; Cool, 4°C	14 days until extraction; 30 days after extraction
Diquat and Paraquat (EPA 549.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$ if Res.Cl., Cool, 4°C,	7days until extraction; 21 days after extraction
Endothall (EPA 548.1)	DW	G, Amber, Teflon-Lined Cap	Cool, 4°C	7 days until extraction; 14 days after extraction
Glyphosate (EPA 547)	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$, Cool, 4°C	14 days
Haloacetic Acids (EPA 552.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L NH_4Cl, Cool, 4°C	14 days until extraction; 7 days after extraction
Semivolatile Organics (EPA 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH≤ 2; Cool, 4°C	14 days until extraction; 30 days after extraction
Nitrosoamines (EPA 521)	DW	G, Amber, Teflon-Lined Cap	Dechlorinate at collection[g] Cool, 4°C	14 days until extraction; 28 days after extraction
Selected Pesticides and Flame Retardants (EPA 527)	DW	G, Amber, Teflon-Lined Cap	See method Cool, 4°C	14 days until extraction; 28 days after extraction
Explosives (EPA 529)	DW	G, Amber, Teflon-Lined Cap	See method Cool, 4°C	14 days until extraction; 30 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Toxicity Characteristic Leaching Procedure (TCLP)				
Semivolatile Organics (EPA 1311/8270)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C, Store in Dark[g] TCLP extract: Cool, 4°C, Store in Dark[g]	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Organochlorine Pesticides (EPA 1311/8081)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Chlorinated Herbicides (EPA 1311/8151)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Mercury (EPA 1311/7470)	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	28 days until extraction; 28 days after extraction
Metals, except Mercury (EPA 1311/6010)	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	180 days until extraction; 180 days after extraction
Volatile Organics (EPA 1311/8260)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C Minimize Headspace TCLP extract: Cool, 4°C, HCl to pH<2, No Headspace	14 days until extraction; 14 days after extraction

a For EPA SW-846 methods the method number is listed generically, without specific revision suffixes.
b DW = Drinking Water, W = Water; S = Soil or Sediment; HW = Hazardous Waste
c P = Polyethylene; G = Glass, F- Fluoropolymer
d For chlorinated water samples
e The maximum holding time is dependent upon the geographical proximity of sample source to the laboratory.
f Fourteen days until extraction for soil, sediment, and sludge samples.
g If the water sample contains residual chlorine, 10% sodium thiosulfate is used to dechlorinate.

Figure 8-1
Chain of Custody Form

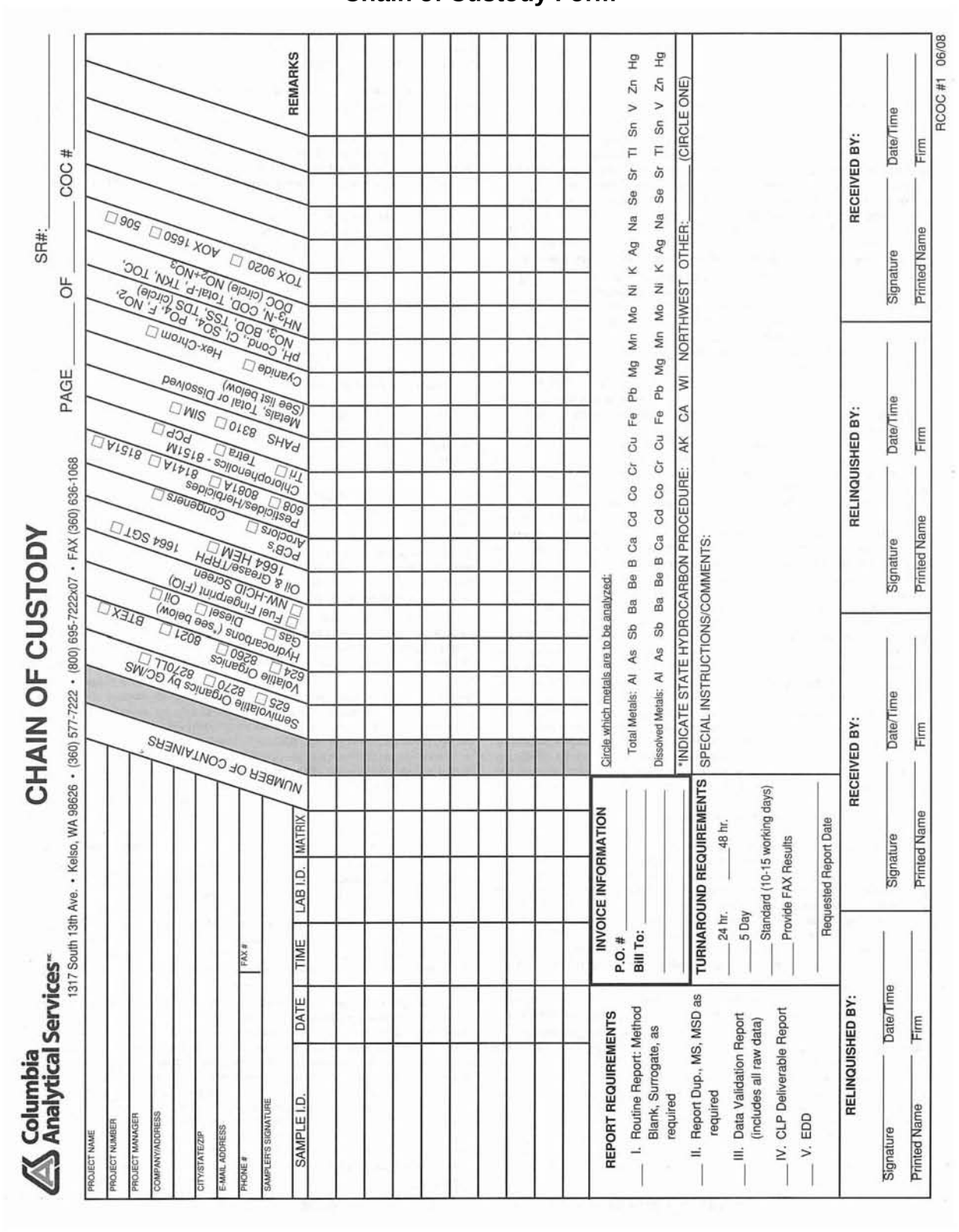


Figure 8-2

Columbia Analytical Services, Inc.
Cooler Receipt and Preservation Form

PC_____

Client / Project:_____ Service Request *K09*_____

Received:_____ Opened:_____ By:_____

1. Samples were received via? ***US Mail*** ***Fed Ex*** ***UPS*** ***DHL*** ***GH*** ***GS*** ***PDX*** ***Courier*** ***Hand Delivered***
2. Samples were received in: (circle) ***Cooler*** ***Box*** ***Envelope*** ***Other***_____ ***NA***
3. Were custody seals on coolers? NA Y N If yes, how many and where?_____
   If present, were custody seals intact? Y N If present, were they signed and dated? Y N
4. Is shipper's air-bill filed? If not, record air-bill number:_____ NA Y N

_____ _____ _____

5. **Temperature of cooler(s) upon receipt (°C):** _____ _____ _____ _____
   Temperature Blank (°C): _____ _____ _____ _____
   Thermometer ID: _____ _____ _____ _____
6. If applicable, list Chain of Custody Numbers: _____ _____ _____ _____
7. Packing material used. ***Inserts*** ***Baggies*** ***Bubble Wrap*** ***Gel Packs*** ***Wet Ice*** ***Sleeves*** ***Other***_____
8. Were custody papers properly filled out (ink, signed, etc.)? NA Y N
9. **Did all bottles arrive in good condition (unbroken)?** *Indicate in the table below.* NA Y N
10. Were all sample labels complete (i.e analysis, preservation, etc.)? NA Y N
11. Did all sample labels and tags agree with custody papers? *Indicate in the table below* NA Y N
12. **Were appropriate bottles/containers and volumes received for the tests indicated?** NA Y N
13. Were the pH-preserved bottles tested* received at the appropriate pH? *Indicate in the table below* NA Y N
14. Were VOA vials and 1631 Mercury bottles received without headspace? *Indicate in the table below.* NA Y N
15. **Are CWA Microbiology samples received with >1/2 the 24hr. hold time remaining from collection?** NA Y N
16. Was C12/Res negative? NA Y N

Sample ID on Bottle	Sample ID on COC	Sample ID on Bottle	Sample ID on COC

Sample ID	Bottle Count	Bottle Type	Out of Temp	Head-space	Broken	pH	Reagent	Volume added	Reagent Lot Number	Initials

*Does not include all pH preserved sample aliquots received. See sample receiving SOP (SMO-GEN).

Additional Notes, Discrepancies, & Resolutions:_____

_____

_____

Page 1 of: *1* *2*

Figure 8-3
Tier V Form

Client :

Project Chemist :

Project Name :

Service Request :

Project Number :

SMO LimsTemplate ID :

Project Description :

QAPP/SOW Information :

Reporting

Tier Level :

PDF:

Report to :

In result field use :

EDD :

Flagging Requirements :

Other Requirements :

Sample Considerations

Sample Limitations :

Sample Prep/Analysis :

Non-Standard Holdtimes :

Historical Data :

Comments :

9.0 ANALYTICAL PROCEDURES

Columbia Analytical employs methods and analytical procedures from a variety of external sources. The primary method references are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IVA, IVB, and online updates for hazardous waste samples, and USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. Complete citations for these references can be found in Section 18.0. Other published procedures, such as state-specific methods, program-specific methods (such as Puget Sound Protocols), or in-house methods may be used. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection limit, the concentration of the analyte being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives. The implementation of methods by Columbia Analytical is described in SOPs specific to each method. A list of NELAP-accredited methods is given in Appendix E. Further details are described below.

9.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks.

Columbia Analytical maintains SOPs for use in both technical and administrative functions. SOPs are written following standardized format and content requirements. Each SOP is reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the Quality Assurance Manager). All SOPs undergo a documented annual review to make sure current practices are described. The QA Manager maintains a comprehensive list of current SOPs. The document control process ensures that only the most currently prepared version of an SOP is being used. The QA Manual, QAPPs, SOPs, standards preparation logbooks, maintenance logbooks, et al., are controlled documents. The procedures for document control are described in the *SOP for Document Control* (ADM-DOC_CTRL). In addition to SOPs, each laboratory department maintains a current file, accessible to all laboratory staff, of the current methodology used to perform analyses. Laboratory notebook entries are standardized following the guidelines in the *SOP for Making Entries into Logbooks and onto Benchsheets* (ADM-DATANTRY). Entries made into laboratory notebooks are reviewed and approved by the appropriate supervisor at a regular interval.

9.2 Deviation from Standard Operating Procedures

When a customer requests a modification to an SOP (such as a change in reporting limit, addition or deletion of target analyte(s), etc.), the project chemist handling that project must discuss the proposed deviation with the department manager in charge of the analysis and obtain their approval to accept the project. The project chemist is responsible for documenting the approved or allowed deviation from the SOP by placing a detailed description of the deviation attached to the quotation or in the project file and also providing an appropriate comment on the service request when the samples are received.

For circumstances when a deviation or departure from company policies or procedures involving any non-technical function is found necessary, approval must be obtained from the appropriate supervisor, manager, the laboratory director, or other level of authority. Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the laboratory director will address the possible need for a change in policy.

9.3 Modified Procedures

Columbia Analytical strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating procedures are available to analysts and are also available to our clients for review, especially those for "Modified" methods. Client approval is obtained for the use of "Modified" methods prior to the performance of the analysis.

9.4 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that Columbia Analytical has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner. The minimum requirements of an analytical batch are:

1) The number of (field) samples in a batch is not to exceed 20.
2) All (field) samples in a batch are of the same matrix.
3) The QC samples to be processed with the (field) samples include:
   a) Method Blank (a.k.a. Laboratory Reagent Blank)

      Function: Determination of laboratory contamination.

   b) Laboratory Control Sample

      Function: Assessment of method performance

   c) Matrix Spiked (field) Sample (a.k.a. Laboratory Fortified Sample Matrix)*

      Function: Assessment of matrix bias

   d) Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample (a.k.a. Laboratory Duplicate)*

      Function: Assessment of batch precision

   * A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked or duplicated.
4) A single lot of reagents is used to process the batch of samples.
5) Each operation within the analysis is performed by a single analyst, technician, chemist, or by a team of analysts/technicians/chemists.

6) Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours.

7) (Field) samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic constituents, it begins when the samples are extracted.

8) The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch (digestion, extraction, etc.), the QC samples in the batch do not require analysis each time a field sample within the preparation batch is analyzed (multiple instrument sequences to analyze all field samples in the batch need not include re-analyses of the QC samples).

9) The batch is to be assigned a unique identification number that can be used to correlate the QC samples with the field samples.

10) Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.

11) Project-specific requirements may be exceptions. If project, program, or method requirements are more stringent than these laboratory minimum requirements, then the project, program, or method requirements will take precedence. However, if the project, program, or method requirements are less stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

9.5 Specialized Procedures

Columbia Analytical not only strives to provide results that are scientifically sound, legally defensible, and of known and documented quality; but also strives to provide the best solution to analytical challenges. Procedures using specialized instrumentation and methodology have been developed to improve sensitivity (provide lower detection limits), selectivity (minimize interferences while maintaining sensitivity), and overall data quality for low concentration applications. Examples are trace-level Mercury and Methylmercury analyses, reductive precipitation metals analysis, specialized GC/MS analyses, LC/MS analyses, and ultra-low level organics analyses (including PAHs, pesticides and PCBs).

9.6 Sample Cleanup

Columbia Analytical commonly employs several cleanup procedures to minimize known common interferences prior to analysis. EPA methods (3620, 3630, 3640, 3660, and 3665) for cleanup of sample extracts for organics analysis are routinely used to minimize or eliminate interferences that may adversely affect sample results and data usability.

10.0 CALIBRATION PROCEDURES AND FREQUENCY

All equipment and instruments used at Columbia Analytical are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below. Calibration verification is performed according to the applicable analytical methodology. Calibration verification procedures and criteria are listed in laboratory Standard Operating Procedures. Documentation of calibration verification is maintained in appropriate reference files.

Records are maintained to provide traceability of reference materials.

Laboratory support equipment (thermometers, balances, and weights) are routinely verified on an annual basis by a vendor accredited to A2LA or ISO/IEC 17025:2005 International Standards. All analytical measurements generated at Columbia Analytical are performed using materials and/or processes that are traceable to a reference material. Metrology equipment (analytical balances, thermometers, etc.) is calibrated using reference materials traceable to the National Institute of Standards and Technology (NIST). These primary reference materials are themselves recertified on an annual basis. Vendors used for metrology support are required to verify compliance to International Standards by supplying the laboratory with a copy of their scope of accreditation.

All sampling containers provided to the client by the laboratory are purchased as precleaned (Level 1) containers, with certificates of analysis available for each bottle type. This information is provided to the client when requested.

Equipment subjected to overloading or mishandling, or has been shown by verification to be defective; is taken out of service until it is repaired. The equipment is placed back in service only after verifying, by calibration, that the equipment performs satisfactorily.

10.1 Temperature Control Devices

Temperatures are monitored and recorded for all of the temperature-regulating support equipment such as sample refrigerators, freezers, and standards refrigerators. Bound record books are kept which contain daily-recorded temperatures, identification and location of equipment, acceptance criteria and the initials of the technician who performed the checks. The procedure for performing these measurements is provided in the *SOP for Support Equipment Monitoring and Calibration (SOP ADM-SEMC).* The SOP also includes the use of acceptance criteria and correction factors.

Where the operating temperature is specified as a test condition (such as ovens, incubators, evaporators) the temperature is recorded on the raw data. All thermometers are identified according to serial number, and the calibration is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. The NIST thermometer is recertified by a vendor accredited to A2LA or ISO/IEC 17025:2005 International Standard on an annual basis.

10.2 Analytical Balances

The calibration of each analytical balance is checked by the user each day of use with three Class S or S-1 weights, which assess the accuracy of the balance at low, mid-level and high levels bracketing the working range. Records are kept which contain the recorded measurements, identification of the balance, acceptance criteria, and the initials of user who performed the check. The procedure for performing these measurements and use of acceptance criteria is described in the SOP ADM-SEMC. The weights are recertified using NIST traceable standards by an accredited metrology organization on an annual basis.

As needed, the balances are recalibrated using the manufacturers recommended operating procedures. Analytical balances are serviced on a semi-annual basis by an accredited metrology organization.

10.3 Water Purification Systems

Columbia Analytical uses two independent water purification systems is designed to produce deionized water meeting method specifications. One system consists of a series of pumps, filters, and resin beds designed to yield deionized water meeting the specifications of ASTM Type II water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Activated carbon filters are also in series with the demineralizers to produce "organic-free" water. A second system consists of pumps, filters, and treatment components designed to yield deionized water meeting the specifications of ASTM Type I water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Following a written SOP, the status of each system is monitored continuously for conductivity and resistivity with an on-line meter and indicator light, and readings recorded daily in a bound record book. The meter accuracy is verified annually. Deionizers are rotated and replaced on a regular schedule. Microbiology water is checked on a daily basis at a point downstream of the purification system at a tap in the laboratory.

10.4 Source and Preparation of Standard Reference Materials

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. All vendors have fulfilled the requirements for ISO 9001 certification and/or are accredited by A_2LA. Columbia Analytical relies on a primary vendor for the majority of its analytical supplies. Consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Supelco, Ultra Scientific, AccuStandard, Chem Services, Inc., Aldrich Chemical Co., Baker, Spex, etc. are examples of the vendors used. Reference material information is recorded in the appropriate logbook(s) and materials are stored under conditions that provide maximum protection against deterioration and contamination. The logbook entry includes such information as an assigned logbook identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. The date that the standard is received in the laboratory is marked on the container. When the reference material is used for the first time, the date of usage and the initials of the analyst are also recorded on the container.

Stock solutions and calibration standard solutions are prepared fresh as often as necessary according to their stability. All standard solutions are properly labeled as to analyte concentration, solvent, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook(s) following the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP No. ADM-DATANTRY). Prior to sample analysis, all calibration reference materials are verified with a second, independent source of the material (see section 11.3.5).

10.5 Inductively Coupled Plasma-Atomic Emission Spectrograph (ICP-AES)

Each emission line on the ICP is calibrated daily against a blank and against standards. Analyses of calibration standards, initial and continuing calibration verification standards, and inter-element interference check samples are carried out as specified in the applicable method SOP and analytical method (i.e. EPA 200.7, 6010B, 6010C, CLP SOW, etc.).

10.6 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)

Each element of interest is calibrated for using a blank and a single standard. Prior to calibration, a short-term stability check is performed on the system. Following calibration, an independent check standard is analyzed, and a continuing calibration verification standard (CCV) is analyzed with every ten samples.

10.7 Atomic Absorption Spectrophotometers (AAS)

These instruments are calibrated daily using a minimum of four standards and a blank. Calibration is validated using reference standards, and is verified at a minimum frequency of once every ten samples. Initial calibration points cannot be “dropped” from the resulting calibration curve.

10.8 GC/MS Systems

All GC/MS instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise) using procedures outlined in Standard Operating Procedures and/or appropriate USEPA method citations. All reference materials used for this function are vendor-certified standards. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method. For isotope dilution procedures, the internal standard response(s) and labeled compound recovery must meet method criteria. Method-specific instrument tuning is regularly checked using bromofluorobenzene (BFB) for volatile organic chemical (VOC) analysis, or decafluorotriphenylphosphine (DFTPP) for semi-volatile analysis. Mass spectral peaks for the tuning compounds must conform both in mass numbers and in relative intensity criteria before analyses can proceed. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.9 Gas Chromatographs and High Performance Liquid Chromatographs

Calibration and standardization follow SOP guidelines and/or appropriate USEPA method citations. All GC and HPLC instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise). The lowest standard is equivalent to the method reporting limit; additional standards define the working

range of the GC or LC detector. Results are used to establish response factors (or calibration curves) and retention-time windows for each analyte. Calibration is verified at a minimum frequency of once every ten samples, unless otherwise specified by the reference method. *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.10 LC/MS Systems

Calibration and tuning procedures are included in analytical SOPs written specifically for these tests. In general, multiple concentration levels for the analytes of interest are used to generate calibration curves. All reference materials used for this function are vendor-certified standards. Calibration and tuning verification is performed at SOP-defined intervals. Any other system performance checks are described in the applicable SOP. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.11 UV-Visible Spectrophotometer (manual colorimetric analyses)

Routine calibrations for colorimetric and turbidimetric analyses involve generating a 5-point calibration curve including a blank. Initial calibration points cannot be “dropped” from the resulting calibration curve. Correlation coefficients must meet method or SOP specifications before analysis can proceed. Independent calibration verification standards (ICVs) are analyzed with each batch of samples. Continuing calibration is verified at a minimum frequency of once every ten samples. Typical UV-Visible spectrophotometric methods at Columbia Analytical include total phenolics, phosphates, surfactants and tannin-lignin.

10.12 Flow Injection Analyzer (automated colorimetric analysis)

A minimum of six standards and a blank are used to calibrate the instrument for cyanide analysis. A blank and (minimum of) five standards are used to calibrate the instrument for all other automated chemistries. Initial calibration points cannot be “dropped” from the resulting calibration curve. Standard Columbia Analytical acceptance limits are used to evaluate the calibration curve prior to sample analysis.

10.13 Ion Chromatographs

Calibration of the ion chromatograph (IC) involves generating a calibration curve with the method-specified number of points (or more). Initial calibration points cannot be “dropped” from the resulting calibration curve. A correlation coefficient of $\geq$ 0.995 for the curve is required before analysis can proceed. Quality Control (QC) samples that are routinely analyzed include blanks and laboratory control samples. The target analytes typically determined by the IC include nitrate, nitrite, chloride, fluoride, sulfate and drinking water inorganic disinfection byproducts. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method.

10.14 Turbidimeter

Calibration of the turbidimeter requires analysis of three Nephelometric Turbidity Unit (NTU) formazin standards. Quality Control samples that are routinely analyzed include blanks, Analytical Products Group® QC samples (or equivalent) and duplicates.

10.15 Ion-selective electrode

The method-prescribed numbers of standards are used to calibrate the electrodes before analysis. The slope of the curve must be within acceptance limits before analysis can proceed. Quality Control samples that are routinely analyzed include blanks, LCSs and duplicates.

10.16 Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following the *SOP Checking Volumetric Labware (ADM-VOLWARE)*. Both accuracy and precision verifications are performed, at intervals applicable to the pipet and use. The results of all calibration verifications are recorded in bound logbooks.

10.17 Other Instruments

Calibration for the total organic carbon (TOC), total organic halogen (TOX), and other instruments is performed following manufacturer's recommendations and applicable SOPs.

11.0 QUALITY CONTROL

A primary focus of Columbia Analytical's Quality Assurance (QA) Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis on field samples, acceptable method performance is established by performing demonstration of capability analyses. Performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. Columbia Analytical has established Quality Control (QC) objectives for precision and accuracy that are used to determine the acceptability of the data that is generated. These QC limits are either specified in the test methodology or are statistically derived based on the laboratory's historical data. Quality Control objectives are defined below.

11.1 Quality Control Objectives

11.1.2 Demonstration of Capability - A demonstration of capability (DOC) is made prior to using any new test method or when a technician is new to the method. This demonstration is made following regulatory, accreditation, or method specified procedures. In general, this demonstration does not test the performance of the method in real world samples, but in the applicable clean matrix free of target analytes and interferences.

A quality control sample material may be obtained from an outside source or may be prepared in the laboratory. The analyte(s) is (are) diluted in a volume of clean matrix (for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples). Where specified, the method-required concentration levels are used. Four aliquots are prepared and analyzed according to the test procedure. The mean recovery and standard deviations are calculated and compared to the corresponding acceptance criteria for precision and accuracy in the test method or laboratory-generated acceptance criteria (if there are not established mandatory criteria). All parameters must meet the acceptance criteria. Where spike levels are not specified, actual Laboratory Control Sample results may be used to meet this requirement, provided acceptance criteria is met.

11.1.3 Accuracy - Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of laboratory-fortified blanks, standard reference materials, and standard solutions. In addition, laboratory-fortified (i.e. matrix-spiked) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value. If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis (e.g., extraction efficiencies) or caused by an artifact of the measurement system (e.g., contamination). Columbia Analytical utilizes several quality control measures to eliminate analytical bias, including systematic analysis of method blanks, laboratory control samples and independent calibration verification standards. Because bias can be positive or negative, and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement

11.1.4 Precision - Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: repeatability - the random error associated with measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions, and reproducibility - the random error associated with measurements made by different test operators, in different laboratories, using the same method but different equipment to analyze identical samples of test material.

"Within-batch" precision is measured using replicate sample or QC analyses and is expressed as the relative percent difference (RPD) between the measurements. The "batch-to-batch" precision is determined from the variance observed in the analysis of standard solutions or laboratory control samples from multiple analytical batches.

11.1.5 Control Limits - The control limits for accuracy and precision originate from two different sources. For analyses having enough QC data, control limits are calculated at the 99% confidence limits. For analyses not having enough QC data, or where the method is prescriptive, control limits are taken from the method on which the procedure is based. If the method does not have stated control limits, then control limits are assigned method-default or reasonable values. Control limits are updated periodically when new statistical limits are generated for the appropriate surrogate, laboratory control sample, and matrix spike compounds (typically once a year) or when method prescribed limits change. The updated limits are reviewed by the Quality Assurance Manager. The new control limits replace the previous limits and data is assessed using the new values. Current acceptance limits for accuracy and precision are available from the laboratory. For inorganics, the precision limit values listed are for laboratory duplicates. For organics, the precision limit values listed are for duplicate laboratory control samples or duplicate matrix spike analyses.

11.1.6 Representativeness - Representativeness is the degree to which the field sample, being properly preserved, free of contamination, and analyzed within holding time, represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed represents the entire field sample submitted for analysis. Columbia Analytical has sample handling procedures to ensure that the sample used for analysis is representative of the entire sample.

These include the *SOP for Subsampling and Compositing of Samples* and the *SOP for Tissue Sample Preparation*. Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample.

11.1.7 Comparability – Comparability expresses the confidence with which one data set can be compared to another and is directly affected by data quality (accuracy and precision) and sample handling (sampling, preservation, etc). Only data of known quality can be compared. The objective is to generate data of known quality with the highest level of comparability, completeness, and usability. This is achieved by employing the quality controls listed below and standard operating procedures for the handling and analysis of all samples. Data is reported in units specified by the client and using Columbia Analytical or project-specified data qualifiers.

11.2 Method Detection Limits and Method Reporting Limits

Method Detection Limits (MDL) for methods performed at Columbia Analytical/Kelso is determined during initial method set up and if any significant changes are made. If an MDL study is not performed annually, the established MDL is verified by performing a limit of detection (LOD) verification on every instrument used in the analysis. The MDLs are determined by following the *SOP for Performing Method Detection Limits Studies and Establishing Limits of Detection and Quantitation (ADM-MDL),* which is based on the procedure in 40 CFR Part 136, Appendix B. As required by NELAP and DoD protocols, the validity of MDLs is verified using LOD verification samples.

The Method Reporting Limit (MRL) is the lowest amount of an analyte in a sample that can be quantitatively determined with stated, acceptable precision and accuracy under stated analytical conditions (i.e. limit of quantitation- LOQ). LOQ are analyzed on an annual basis and cannot be lower than the lowest calibration standard. Current MDLs and MRLs are available from the laboratory.

11.3 Quality Control Procedures

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures and listed below. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below.

11.3.1 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is an analyte-free matrix (water, soil, etc.) subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute is used. The method blank is analyzed to demonstrate that the analytical system itself does not introduce contamination. The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, < ½ MRL for the analyte(s) being tested. Otherwise, corrective action must be taken. A method blank is included with the analysis of every sample preparation batch, every 20 samples, or as stated in the method, whichever is more frequent.

11.3.2 Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve. Additional project-specific requirements may also apply to calibration blanks.

11.3.3 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of either analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed. The frequency of CCB analysis is either once every ten samples or as indicated in the method, whichever is greater. Additional project-specific requirements may also apply to continuing calibration blanks.

11.3.4 Calibration Standards

Calibration standards are solutions of known concentration prepared from primary standard or stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

11.3.5 Initial (or Independent) Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are standards that are analyzed *after* calibration with newly prepared standard(s) but *prior to* sample analysis, in order to verify the validity and accuracy of the standards used in the calibration. Once it is determined that there is no reference material defect or systematic error in preparation of the calibration standard(s), standards are considered valid and may be used for subsequent calibrations and quantitative determinations (as expiration dates and methods allow). The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards ("second-source"). ICVs are also analyzed in accordance with method-specific requirements.

11.3.6 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

11.3.7 Internal Standards

Internal standards are known amounts of specific compounds that are added to each sample prior to instrument analysis. Internal standards are generally used for GC/MS and ICP-MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by matrix effects. The requirements for evaluation of internal standards are specified in each method and SOP.

11.3.8 Surrogates

Surrogates are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate, and the recovery is a measurement of the overall method performance.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

11.3.9 Laboratory Control Samples

The laboratory control sample (LCS) is an aliquot of analyte-free water or analyte-free solid (or anhydrous sodium sulfate or equivalent) to which known amounts of the method analyte(s) is (are) added. A reference material of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent. The LCS sample is prepared and analyzed in exactly the same manner as the field samples.

The percent recovery of the target analytes in the LCS is compared to established control limits and assists in determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

11.3.10 Laboratory Fortified Blanks - LFB

A laboratory blank fortified at the MRL used to verify the minimum reporting limit. The LFB is carried through the entire extraction and analytical procedure. A LFB is required with every batch of drinking water samples.

11.3.11 Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix)

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) is (are) added. The samples are then prepared and analyzed in

the same analytical batch, and in exactly the same manner as are routine samples. For the appropriate methods, matrix spiked samples are prepared and analyzed and at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

$$Recovery\ (\%) = (S - A) \times 100 \div T$$

Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

11.3.12 Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed. The relative percent difference between duplicate analyses or between an MS and DMS is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

$$Relative\ Percent\ Difference\ (RPD) = (S1 - S2) \times 100 \div S_{ave}$$

Where S1 and S2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and

S_{ave} = The average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, either duplicates (and/or matrix spikes) or MS/DMS analyses are performed at a minimum frequency of one set per 20 samples. If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

11.3.13 Interference Check Samples

An interference check sample (ICS) is a solution containing both interfering and analyte elements of known concentration that can be analyzed to verify background and interelement correction factors in metals analyses. The ICS is prepared to contain known concentrations (method or program specific) of elements that will provide an adequate test of the correction factors. The ICS is analyzed at the beginning and end of an analytical run or at a method-specified frequency. Results must meet method criteria and any project-specific criteria.

11.3.14 Post Digestion Spikes

Post digestion spikes are samples prepared for metals analyses that have an analyte spike added to determine if matrix effects may be a factor in the results. The spike addition should produce a method-specified minimum concentration above the method reporting limit. A post digestion spike is analyzed with each batch of samples and recovery criteria are specified for each method.

11.3.15 Control Charting

The generation of control charts is routinely performed at Columbia Analytical. Surrogate, Matrix Spike and LCS recoveries are all monitored and charted. In addition, the laboratory also monitors the Relative Percent Difference (RPD) measurement of precision. Control charts are available to each individual laboratory unit to monitor the data generated in its facility using control charts that have been programmed to identify various trends in the analytical results. If trends in the data are perceived, various means of corrective action may then be employed in order to prevent future problems with the analytical system(s). Finally, data quality reports using control charts are generated for specific clients and projects pursuant to contract requirements. The control charting procedure is described in the SOP for *Control Charting Quality Control Data* (ADM-CHRT).

11.3.16 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at Columbia Analytical undergoes a rigorous cleansing procedure prior to every usage. A number of SOPs have been generated that outline the various procedures used at Columbia Analytical; each is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

12.0 DATA REDUCTION, VALIDATION, AND REPORTING

Columbia Analytical reports the analytical data produced in its laboratories to the client via the certified analytical report (CAR). This report includes a transmittal letter, a case narrative, client project information, specific test results, quality control data, chain of custody information, and any other project-specific support documentation. The following procedures describe our data reduction, validation and reporting procedures.

12.1 Data Reduction and Review

Results are generated by the analyst who performs the analysis and works up the data. All data is initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation, etc.). Equations used for calculation of results are found in the applicable analytical SOPs. The resulting data set is either manually entered (e.g., titrimetric or microbiological data) into an electronic report form or is electronically transferred into the report from the software used to process the original data set (e.g., chromatographic software). Once the complete data set has been transferred into the proper electronic report form(s), it is then printed. The resulting hardcopy version of the electronic report is then reviewed by the analyst for accuracy. Once the primary analyst has checked the data for accuracy and acceptability, the hardcopy is forwarded to the supervisor or second qualified analyst, who reviews the data for errors. Where calculations are not performed using a validated software system, the reviewer rechecks a minimum of 10% of the calculations. When the entire data set has been found to be acceptable, a final copy of the report is printed and signed by the laboratory supervisor, departmental manager or designated laboratory staff. The entire data package is then placed into the appropriate service request file, and an electronic copy of the final data package is forwarded to the appropriate personnel for archival. Data review procedures are described in the *SOP for Laboratory Data Review Process.*

Policies and procedures for manual editing of data are established. The analyst making the change must initial and date the edited data entry, without obliteration of the original entry. The policies and procedures are described in the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP ADM-DATANTRY).

Policies and procedures for electronic manual integration of chromatographic data are established. The analyst performing the integration must document the integration change by printing both the “before” and “after” integrations and including them in the raw data records. The policies and procedures are described in the *SOP for Manual Integration of Chromatographic Peaks* (SOP ADM-INT).

12.2 Confirmation Analysis

12.2.1 Gas Chromatographic and Liquid Chromatographic Analyses

For gas chromatographic (GC) and liquid chromatographic (LC) analyses, all positive results are confirmed by a second column, a second detector, a second wavelength (HPLC/UV), or by GC/MS analysis, unless exempted by one of the following situations:

- The analyte of interest produces a chromatogram containing multiple peaks exhibiting a characteristic pattern, which matches appropriate standards. This is limited to petroleum hydrocarbon analyses (e.g., gasoline and diesel) and does not include polychlorinated biphenyls.
- The sample meets all of the following requirements:
    1. All samples (liquid or solid) come from the same source (e.g., groundwater samples from the same well) for continuous monitoring. Samples of the same matrix from the same site, but from different sources (e.g., different sampling locations) are not exempt.
    2. All analytes have been previously analyzed in sample(s) from the same source (within the last year), identified and confirmed by a second column or by GC/MS. The chromatogram is largely unchanged from the one for which confirmation was carried out. The documents indicating previous confirmation must be available for review.

12.2.2 Confirmation Data

Confirmation data will be provided as specified in the method. Identification criteria for GC, LC or GC/MS methods are summarized below:

- GC and LC Methods
    1. The analyte must fall within plus or minus three times the standard deviation (established for the analyte/column) of the retention time of the daily midpoint standard in order to be qualitatively identified. The retention-time windows will be established and documented, as specified in the appropriate Standard Operating Procedure (SOP).
    2. When sample results are confirmed by two dissimilar columns or detectors, the agreement between quantitative results must be evaluated. The relative percent difference between the two results is calculated and evaluated against SOP and/or method criteria.
- GC/MS Methods - Two criteria are used to verify identification:
    1. Elution of the analyte in the sample will occur at the same relative retention time (RRT) as that of the analyte in the standard.
    2. The mass spectrum of the analyte in the sample must, in the opinion of a qualified analyst or the department manager, correspond to the spectrum of the analyte in the standard or the current GC/MS reference library.

12.3 Data Review and Validation of Results

The integrity of the data generated is assessed through the evaluation of the sample results, calibrations, and QC samples (method blanks, laboratory control samples, sample duplicates, matrix spikes, trip blanks, etc.). A brief description of the evaluation of these analyses is described below, with details listed in applicable SOPs. The criteria for evaluation of QC samples are listed within each method-specific SOP. Other data evaluation measures may include (as necessary) a check of the accuracy check of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed.

Note: Within the scope of this document, all possible data assessment requirements for various project protocols cannot be included in the listing below. This listing gives a general description of data evaluation practices used in the laboratory in compliance with NELAP Quality Systems requirements. Additional requirements exist for certain programs, such as projects under the DoD QSM protocols, and project-specific QAPPs.

- Method Calibration – Following the analysis of calibration blanks and standards according to the applicable SOP the calibration correlation coefficient, average response factor, etc. is calculated and compared to specified criteria. If the calibration meets criteria analysis may continue. If the calibration fails, any problems are isolated and corrected and the calibration standards reanalyzed. Following calibration and analysis of the independent calibration verification standard(s) the percent difference for the ICV is calculated. If the percent difference is within the specified limits the calibration is complete. If not, the problem associated with the calibration and/or ICV are isolated and corrected and verification and/or calibration is repeated.
- Continuing Calibration Verification (CCV) – Following the analysis of the CCV standard the percent difference is calculated and compared to specified criteria. If the CCV meets the criteria analysis may continue. If the CCV fails, routine corrective action is performed and documented and a 2nd CCV is analyzed. If this CCV meets criteria, analysis may continue, including any reanalysis of samples that were associated with a failing CCV. If the routine corrective action failed to produce an immediate CCV within criteria, then either acceptable performance is demonstrated (after additional corrective action) with two consecutive calibration verifications or a new initial calibration is performed.
- Method Blank – Results for the method blank are calculated as performed for samples. If results are less than the MRL (<½ MRL for DoD projects), the blank may be reported. If not, associated sample results are evaluated to determine the impact of the blank result. If possible, the source of the contamination is determined. If the contamination has affected sample results the blank and samples are reanalyzed. If positive blank results are reported, the blank (and sample) results are flagged with an appropriate flag, qualifier, or footnote.
- Sample Results (Inorganic) – Following sample analysis and calculations (including any dilutions made due to the sample matrix) the result is verified to fall within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. When sample and sample duplicates are analyzed for precision, the calculated RPD is compared to the specified limits. The sample and duplicate are reanalyzed if the criteria are exceeded. The samples may require re-preparation and reanalysis. For metals, additional measures as described in the applicable SOP may be taken to further evaluate results (dilution tests and/or post-digestion spikes). Results are reported when within the calibration range, or as estimates when outside the calibration range. When dilutions are

performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL's including alternative analysis.

- Sample Results (Organic) – For GC/MS analyses, it is verified that the analysis was within the prescribed tune window. If not, the sample is reanalyzed. Following sample analysis and calculations (including any dilutions made due to the sample matrix) peak integrations, retention times, and spectra are evaluated to confirm qualitative identification. Internal standard responses and surrogate recoveries are evaluated against specified criteria. If internal standard response does not meet criteria, the sample is diluted and reanalyzed. Results outside of the calibration range are diluted to within the calibration range. For GC and HPLC tests, results from confirmation analysis are evaluated to confirm positive results and to determine the reported value. The procedure to determine which result to report is described in the SOP *Confirmation Procedure for GC and HPLC Analysis (SOC-CONF).* If obvious matrix interferences are present, additional cleanup of the sample using appropriate procedures may be necessary and the sample is reanalyzed. When dilutions are performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL's including additional cleanup.
- Surrogate Results (Organic) – Following sample analysis and data reduction, the percent recovery of each surrogate is compared to specified control limits. If recoveries are acceptable, the results are reported. If recoveries do not fall within control limits, the sample matrix is evaluated. When matrix interferences are present or documented, the results are reported with a qualifier that matrix interferences are present. If no matrix interferences are present and there is no cause for the outlier, the sample is reprepared and reanalyzed. However, if the recovery is above the upper control limit with non-detected target analytes, the sample may be reported. All surrogate recovery outliers are appropriately qualified on the report.
- Duplicate Sample and/or Duplicate Matrix Spike Results – The RPD is calculated and compared to the specified control limits. If the RPD is within the control limits the result is reported. If not, an evaluation of the sample is made to verify that a homogenous sample was used. Despite the use of homogenizing procedures prior to sample preparation or analysis, the sample may not be homogenous or duplicate sample containers may not have been sample consistently. If non-homogenous, the result is reported with a qualifier about the homogeneity of the sample. Also, the results are compared to the MRL. If the results are less than five times the MRL, the results are reported with a qualifier that the high RPD is due to the results being near the MRL. If the sample is homogenous and results above five times the MRL, the samples and duplicates are reanalyzed. If re-analysis also produces out-of-control results, the results are reported with an appropriate qualifier.
- Laboratory Control Sample Results – Following analysis of the LCS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits, the analysis is in control and results may be reported. If not, this indicates that the analysis is not in control. Samples associated with the 'out of control' LCS, shall be considered suspect and the samples re-extracted or re-analyzed or the data reported with the appropriate qualifiers. For analysis where a large number of analytes are in the LCS, it becomes more likely that some analytes (marginal exceedences) will be outside the control limits. The procedure described in the 2003 NELAC standards, Appendix D.1.1.2.1 are used to determine if the LCS is effective in validating the analytical system and the associated samples.

- Matrix Spike Results – Following analysis of the MS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits the results may be reported. If not, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. It is verified that the spike level is at least five times the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as performing any additional cleanups, dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

12.4 Data Reporting

When an analyst determines that a data package has met the data quality objectives (and/or any client-specific data quality objectives) of the method and has qualified any anomalies in a clear, acceptable fashion, the data package is reviewed by a trained chemist. Prior to release of the report to the client, the project chemist reviews and approves the entire report for completeness and to ensure that any and all client-specified objectives were successfully achieved. The original raw data, along with a copy of the final report, is filed in project files by service request number for archiving. Columbia Analytical maintains control of analytical results by adhering to standard operating procedures and by observing sample custody requirements. All data are calculated and reported in units consistent with project specifications, to enable easy comparison of data from report to report.

To the extent possible, samples shall be reported only if all QC measures are acceptable. If a QC measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). The *SOP for Data Reporting and Report Generation* addresses the flagging and qualification of data. The Columbia Analytical-defined data qualifiers, state-specific data qualifiers, or project-defined data qualifiers are used depending on project requirements. A case narrative may be written by the project chemist to explain problems with a specific analysis or sample, etc.

For subcontracted analyses, the Project Chemist verifies that the report received from the subcontractor is complete. This includes checking that the correct analyses were performed, the analyses were performed for each sample as requested, a report is provided for each analysis, and the report is signed. The Project Chemist accepts the report if all verification items are complete. Acceptance is demonstrated by forwarding the report to the Columbia Analytical client.

12.5 Documentation

Columbia Analytical maintains a records system which ensures that all laboratory records of analysis data retained and available. Analysis data is retained for 5 years from the report date unless contractual terms or regulations specify a longer retention time. The archiving system is described in the *SOP for Data Archiving*.

12.5.1 Documentation and Archiving of Sample Analysis Data

The archiving system includes the following items for each set of analyses performed:

- Benchsheets describing sample preparation (if appropriate) and analysis;
- Instrument parameters (or reference to the data acquisition method);
- Sample analysis sequence;
- Instrument printouts, including chromatograms and peak integration reports for all samples, standards, blanks, spikes and reruns;
- Logbook ID number for the appropriate standards;
- Copies of report sheets submitted to the work request file; and
- Copies of Nonconformity and Corrective Action Reports, if necessary.

Individual sets of analyses are identified by analysis date and service request number. Since many analyses are performed with computer-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed, they are written on the integration report or securely stapled to the chromatogram, if done on a separate sheet.

For organics analysis, data applicable to all analyses within the batch, such as GCMS tunes, CCVs, batch QC, and analysis sequences; are kept using a separate documentation system. This system is used to archive data on a batch-specific basis and is segregated according to the date of analysis. This system also includes results for the most recent calibration curves, as well as method validation results.

12.6 Deliverables

In order to meet individual project needs, Columbia Analytical provides several levels of analytical reports. Standard specifications for each level of deliverable are described in Table 12-1. Variations may be provided based on client or project specifications. This includes (but is not limited to) to following specialized deliverables:

- ADEC – Alaska Department of Conservation specified data package
- ACOE/HTRW – Army Corps of Engineers specified data package and reporting requirements (HTRW, CERP, FUDS, etc.)
- AFCEE – Air Force Center for Environmental Excellence project-specific reporting

When requested, Columbia Analytical provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. Columbia Analytical is capable of generating EDDs with many different formats and specifications. The EDD is prepared by report production staff using the electronic version of the laboratory report to minimize transcription errors. User guides and EDD specification outlines are used in preparing the EDD. The EDD is reviewed and compared to the hard-copy report for accuracy.

Table 12-1
Descriptions of Columbia Analytical Standard Data Deliverables

Tier I. Routine Certified Analytical Report (CAR) includes the following:

1. Transmittal letter
2. Sample analytical results
3. Method blank results
4. Surrogate recovery results and acceptance criteria for applicable organic methods
5. Chain of custody documents
6. Dates of sample preparation and analysis for all tests

Tier II and IIA. In addition to the Tier I Deliverables, this CAR includes the following:

1. Matrix spike result(s) with calculated recovery and including associated acceptance criteria
2. Duplicate or duplicate matrix spike result(s) (as appropriate to method), with calculated relative percent difference
3. Tier IIA also includes Laboratory Control Sample (LCS) result(s) with calculated recovery and including associated acceptance criteria

Tier III. Data Validation Package. In addition to the Tier II Deliverables, this CAR includes the following:

1. Case narrative
2. Calibration records and results of initial and continuing calibration verification standards, with calculated recoveries
3. Results of laboratory control sample (LCS) or Quality Control check sample, with calculated recovery and/or associated acceptance limit criteria
4. Results of calibration blanks or solvent blanks (as appropriate to method)
5. Summary forms for associated QC and calibration parameters
6. Copies of all raw data, including extraction/preparation bench sheets, chromatograms, and instrument printouts. For GC/MS, this includes tuning criteria and mass spectra of all positive hits. Results and spectra of TIC compounds will be included upon request.

Tier IV. CLP-Level Data Validation Package.

A complete Data Validation Package containing all sample results, quality control and calibration results, and raw data necessary to fulfill all deliverable requirements of an EPA Contract Laboratory Program (CLP) data package.

13.0 PERFORMANCE AND SYSTEM AUDITS

Quality audits are an essential part of Columbia Analytical/Kelso's quality assurance program. There are two types of audits used at the facility: System Audits are conducted to qualitatively evaluate the operational details of the QA program, while Performance Audits are conducted by analyzing proficiency testing samples in order to quantitatively evaluate the outputs of the various measurement systems.

13.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of Columbia Analytical/Kelso are conducted regularly by various regulatory agencies and clients. Table 13-1 summarizes some of the major programs in which Columbia Analytical/Kelso participates. Programs and certifications are added as required. Additionally, internal system audits of Columbia Analytical/Kelso are conducted regularly under the direction of the Quality Assurance Manager. The internal audit procedures are described in the *SOP for Internal Audits*. The internal audits are performed as follows:

- Comprehensive lab-wide system audit – performed annually. This audit is conducted such that systems, technical operations, hardcopy data, and electronic data are assessed.
- Hardcopy report audits – minimum of 3 per quarter.
- Electronic audit trail reviews – each applicable instrument per quarter.

All audit findings, and corrective actions are documented. The results of each audit are reported to the Laboratory Director and Department Managers for review. Any deficiencies identified are summarized in the audit report. Managers must respond with corrective actions correcting the deficiency within a defined timeframe. Should problems impacting data quality be found during an internal audit, any client whose data is adversely impacted will be given written notification within the corrective action period (if not already provided).

Electronic data audits may be performed in conjunction with hardcopy data audits. The electronic audits focus on organic chromatographic data and include an examination of audit trails, peak integrations, calibration practices, GCMS tuning data, peak response data, use of appropriate files, and other components of the analysis. The audit also verifies that the electronic data supports the hardcopy reported data.

Additional internal audits or data evaluations may be performed as needed to address any potential data integrity issues that may arise.

13.2 Performance Audits

Columbia Analytical/Kelso also participates in the analysis of interlaboratory proficiency testing (PT) samples. Participation in PT studies is performed on a regular basis and is designed to evaluate all analytical areas of the laboratory. Columbia Analytical routinely participates in the following studies:

- Water Pollution (WP) and additional water parameters, 2 per year.
- Water Supply (WS) PT studies, 2 per year.
- Hazardous Waste/Soil PT studies, 2 per year.
- Underground Storage Tank PT studies, 2 per year.
- Microbiology (WS and WP) PT studies, 2 per year.
- Other studies as required for specific certifications, accreditations, or validations.

PT samples are processed by entering them into the LIMS system as samples (assigned Service Request, due date, testing requirements, etc.) and are processed the same as field samples. The laboratory sections handle samples the same as field samples, performing the analyses following method requirements and performing data review. The laboratory sections submit results to the QA Manager for subsequent reporting to the appropriate agencies or study provider. Results of the performance evaluation samples and audits are reviewed by the Quality Assurance Manager, Laboratory Director, the laboratory staff, and the Columbia Analytical Quality Assurance Director. For any results outside acceptance criteria, the analysis data is reviewed to identify a root cause for the deficiency, and corrective action is taken and documented through nonconformity (NCAR) procedures.

Table 13-1
Current Columbia Analytical Performance and System Audit Programs

Federal and National Programs

- The TNI (The NELAC Institute) National Environmental Laboratory Accreditation Program (NELAP) Accredited Drinking Water, Non-Potable Water, Solid & Hazardous Waste, and Biological Tissue Laboratory
- ANSI-ASQ National Accreditation Board/ACLASS ISO 17025:2005
- DoD- ELAP Environmental Laboratory Accreditation Program
- Naval Facilities Engineering Service Center
  Validated Laboratory for NFESC Parameters
- U.S. Army Corps of Engineers
  Approved Laboratory for USACE Projects
- U.S. EPA Region 8
  Approved Drinking Water Laboratory

State and Local Programs

- State of Alaska, Department of Environmental Conservation
  UST Laboratory, Lab I.D. UST040
- State of Arizona, Department of Health Services
  License No. AZ0339
- State of Arkansas, Department of Environmental Quality
  Certified Environmental Laboratory, Lab I.D. 88-0637
- State of California, Department of Health Services, Environmental Laboratory Accreditation Program
  Certification No. 2286
- State of Colorado, Department of Public Health and Environment
  Certified Drinking Water Laboratory
- State of Florida, Department of Health
  Primary NELAP Accreditation No. E87412
- State of Georgia, Department of Natural Resources
  Certified Drinking Water Laboratory
- State of Hawaii, Department of Health
  Certified Drinking Water Laboratory
- State of Idaho, Department of Health and Welfare
  Certified Drinking Water Laboratory
- State of Indiana, Department of Health
  Certified Drinking Water Laboratory, Lab I.D. C-WA-01
- State of Louisiana, Department of Environmental Quality
  Accredited Environmental Laboratory, Lab I.D. 3016
- State of Louisiana, Department of Health and Hospitals
  Accredited Drinking Water Laboratory, Lab I.D. LA080001
- State of Maine, Department of Human Services
  Certified Environmental Laboratory, Lab I.D. WA0035
- State of Michigan, Department of Environmental Quality
  Certified Drinking Water Laboratory, Lab I.D. 9949

Table 13-1 (continued)

State and Local Programs (continued)

- State of Minnesota, Department of Health
  Certified Environmental Laboratory, Lab I.D. 053-999-368
- State of Montana, Department of Health and Environmental Sciences
  Certified Drinking Water Laboratory, Lab I.D. 0047
- State of Nevada, Division of Environmental Protection
  Certified Drinking Water Laboratory, Lab I.D. WA35
- State of New Jersey, Department of Environmental Protection
  Accredited Environmental Laboratory, Lab I.D. WA005
- State of New Mexico, Environment Department
  Certified Drinking Water Laboratory
- State of North Carolina, Department of Environment and Natural Resources
  Certified Environmental Laboratory, Lab I.D. 605
- State of Oklahoma, Department of Environmental Quality
  General Water Quality/Sludge Testing, Lab I.D. 9801
- State of Oregon, ORELAP Laboratory Accreditation Program
  Accredited Environmental Laboratory, Lab I.D. WA200001
- State of South Carolina, Department of Health and Environmental Control
  Certified Environmental Laboratory, Lab I.D. 61002
- State of Utah, Department of Health, Division of Laboratory Services
  Accredited Environmental Laboratory
- State of Washington, Department of Ecology, Environmental Laboratory Accreditation Program
  Accreditation No. C1203
- State of Wisconsin, Department of Natural Resources
  Accredited Environmental Laboratory, Lab I.D. 998386840

14.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a crucial element of the Quality Assurance program. Instruments at Columbia Analytical (e.g., ICP/MS and ICP systems, GC/MS systems, atomic absorption spectrometers, analytical balances, gas and liquid chromatographs, etc.) are maintained under commercial service contracts or by qualified, in-house personnel. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at Columbia Analytical contain extensive information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on every instrument used at Columbia Analytical before it maybe used for sample analysis. If an instrument is modified or repaired, a return to analytical control is required before subsequent sample analyses can occur. When an instrument is acquired at the laboratory, the following information is noted in a bound maintenance notebook specifically associated with the new equipment:

- The equipment's serial number;
- Date the equipment was received;
- Date the equipment was placed into service;
- Condition of equipment when received (new, used, reconditioned, etc.); and
- Prior history of damage, malfunction, modification or repair (if known).

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at Columbia Analytical. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase.

Responsibility for ensuring that routine maintenance is performed lies with the section supervisor. The supervisor may perform the maintenance or assign the maintenance task to a qualified bench level analyst who routinely operates the equipment. In the case of non-routine repair of capital equipment, the section supervisor is responsible for providing the repair, either by performing the repair themselves with manufacturer guidance or by acquiring on-site manufacturer repair. Each laboratory section maintains a critical parts inventory. The parts inventories include the items needed to perform the preventive maintenance procedures listed in Appendix D.

This inventory or “parts list” also includes the items needed to perform any other routine maintenance and certain in-house non-routine repairs such as gas chromatography/mass spectrometry jet separators and electron multipliers and ICP/MS nebulizer. When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem;
- Repairs and/or maintenance performed;
- Description and/or part number of replaced parts;
- Source(s) of the replaced parts;
- Analyst's signature and date; and
- Demonstration of return to analytical control.

See the table in Appendix D for a list of preventive maintenance activities and frequency for each instrument.

15.0 CORRECTIVE ACTION

Nonconforming events such as errors, deficiencies, deviations from SOP, proficiency (PT) failure or results that fall outside of established QC limits are documented using a *Nonconformity and Corrective Action Report* form. The laboratorys procedure and responsibilities for addressing nonconforming work is defined in the SOP ADM-CA *Corrective Action.*

The laboratory takes all appropriate steps necessary to ensure all sample results are reported with acceptable quality control results. When sample results do not conform to established quality control procedures, responsible management will evaluate the significance of the nonconforming work and take corrective action to address the nonconformance.

If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). Failure to meet established analytical controls, such as the quality control objectives outlined in Section 11, prompts corrective action. In general, corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the supervisor, team leader, the department manager, and/or the Quality Assurance Manager may examine and pursue alternative solutions. In addition, the appropriate project chemist is notified in order to ascertain if the client needs to be notified.

In the event that analyses produce nonconformances with data or results, the problem and the corresponding corrective actions taken are documented on a *Nonconformity and Corrective Action Report* (See Figure 15-1) following the requirements in the *SOP for Corrective Action* (SOP No. ADM-CA). This form is utilized to determine the root cause of the nonconformity and to document corrective actions in response to out-of-control situations. The Quality Assurance Manager reviews each problem, ensuring that appropriate corrective action has been taken by the appropriate personnel. The Nonconformity and Corrective Action Report (NCAR) is filed in the associated service request file and a copy is kept by the Quality Assurance Manager. The Quality Assurance Manager periodically reviews all NCARs looking for chronic, systematic problems that need more in-depth investigation and alternative corrective action consideration. In addition, the appropriate project chemist is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

In addition to internal communication of data issues, the laboratory also maintains a system for dealing with customer complaints. The person who initially receives the feedback (typically the project chemist) is responsible for documenting the complaint. If the project chemist is unable to satisfy the customer, the complaint is brought to the attention of the Client Services Manager, Laboratory Director, or QA Manager for final resolution. The complaint and resolution are documented. The procedure is described in the *SOP for Handling Customer Feedback* (ADM-FDBK).

Figure 15-1

Nonconformity and Corrective Action Report

NCAR No: *Assigned by QA*

PROCEDURE (SOP or METHOD): EVENT DATE: _____

EVENT:
- ☐ Missed Holding Time
- ☐ Method Blank Contamination
- ☐ Equipment Failure
- ☐ SOP Deviation
- ☐ QC Failure
- ☐ Login Error
- ☐ Unacceptable PT Sample Result
- ☐ Other (describe):
- ☐ Lab Error (spilled sample, spiking error, etc.)
- ☐ Project Management Error

INCLUDE NUMBER OF SAMPLES / PROJECTS / CUSTOMERS / SYSTEMS AFFECTED

DETAILED DESCRIPTION

ORIGINATOR: _____ DATE: _____

PROJECT MANAGER(S): _____ NOTIFIED BY: _____ DATE: _____

ROOT CAUSE OF NON-CONFORMITY (POTENTIAL CAUSES COULD BE TRAINING, COMMUNICATION, SPECIFICATIONS, EQUIPMENT, KNOWLEDGE)

What is the cause of the error or finding:

CORRECTIVE ACTION AND OUTCOME

Re-establishment of conformity must be demonstrated and documented. Describe the steps that were taken, or are planned to be taken, to correct the particular Nonconformity and prevent its reoccurrence. Include Project Manager Instructions here.

Is the data to be flagged in the Analytical Report with an appropriate qualifier? ☐ **No** ☐ **Yes**

APPROVAL AND NOTIFICATION

Supervisor Verification and Approval of Corrective Action _____ Date: _____
Comments:

QA PM Verification and Approval of Corrective Action _____ Date: _____
Comments:

Project Manager Verification and Approval of Corrective Action _____ Date: _____
Comments:

Customer Notified by ☐ Telephone ☐ Fax ☐ E-mail ☐ Narrative ☐ Not notified

(Attach record or cite reference where record is located.)

16.0 QUALITY ASSURANCE REPORTS

Quality assurance requires an active, ongoing commitment by Columbia Analytical personnel at all levels of the organization. Communication and feedback mechanisms are designed so that analysts, supervisors and managers are aware of QA issues in the laboratory. Analysts performing routine testing are responsible for generating a data quality narrative or data review document with every analytical batch processed. This report also allows the analyst to provide appropriate notes and/or a narrative if problems were encountered with the analyses. A Non-Conformity and Corrective Action Report (NCAR) (see Section 15.0) may also be attached to the data prior to review. Supervisors or qualified analysts review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and addressed.

It is the responsibility of each laboratory unit to provide the project chemist with a final report of the data, accompanied by signature approval. Footnotes and/or narrative notes must accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate project chemist, who in turn reviews the entire collection of analytical data for completeness and to ensure that any and all client-specified objectives were successfully achieved. A case narrative is written by the project chemist to explain any unusual problems with a specific analysis or sample, etc.

The Quality Assurance Manager (QAM) provides overview support to the project chemists as required (e.g., contractually specified, etc.). The QAM is also responsible for the oversight of all internal and external audits, for all proficiency testing sample and analysis programs, and for all laboratory certification/accreditation responsibilities. The QAM provides the Laboratory Director with quarterly reports that summarize the various QA/QC activities that occurred during the previous quarter. The report addresses such topics as the following:

- Status, schedule, and results of internal and external audits;
- Status, schedule, and results of internal and external proficiency testing studies;
- Status of certifications, accreditations, and approvals;
- Status of QA Manual and SOP review and revision;
- Status of MDLs studies;
- Discussion of QC problems in the laboratory;
- Discussion of corrective action program issues;
- Status of staff training and qualification; and
- Other topics as appropriate.

The Laboratory Director also performs an annual management review of the quality and management systems to identify any necessary changes or improvements to the quality system or quality assurance policies. This review is documented in a report *Management Quality System and Testing Review* and sent to senior management.

17.0 PERSONNEL TRAINING

Technical position descriptions are available for all employees, regardless of position or level of seniority. These documents are maintained by the Human Resources personnel and are available for review. In order to assess the technical capabilities and qualifications of a potential employee, all candidates for employment at Columbia Analytical are evaluated, in part, against the appropriate technical description.

Training begins the first day of employment at Columbia Analytical when the company policies are presented and discussed. Safety and QA/QC requirements are integral parts of all technical SOPs and, consequently, are integral parts of all training processes at Columbia Analytical. Safety training begins with the reading of the *Environmental Health and Safety Manual.* Employees are also required to attend periodic safety meetings where additional safety training may be performed by the Environmental, Health and Safety Officer.

Employees are responsible for complying with the requirements of the QA Manual and QA/QC requirements associated with their function(s). Quality Systems training begins with Quality Assurance orientation for new employees and reading the *Quality Assurance Manual.* During the employees first year, the employee attends *Core Ethics* training and learns about Columbia Analytical Services quality systems. Each employee participates in annual *Ethics Refresher* training, which is part of the Columbia Analytical Improper Practices Prevention Program.

Columbia Analytical also encourages its personnel to continue to learn and develop new skills that will enhance their performance and value to the Company. Ongoing training occurs for all employees through a variety of mechanisms. The “CAS University” education system, external and internal technical seminars and training courses, and laboratory-specific training exercises are all used to provide employees with professional growth opportunities.

All technical training is documented and records are maintained in the QA department. Training requirements and its documentation are described in the SOP (ADM-TRANDOC) *Documentation of Training.* A training plan is developed whenever an employee starts a new procedure to new position. The training plan includes a description of the step-by-step process for training an employee and for initial demonstration of capability. Where the analyst performs the entire procedure, a generic training plan may be used.

17.1 Initial Demonstration of Capability (IDOC)

Training in analytical procedures typically begins with the reading of the Standard Operating Procedure (SOP) for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the applicable Demonstration of Capability analysis qualifies the analyst to perform the method independently. Demonstration of Capability is performed by one of the following:

- Successful completion of an Initial Precision and Recovery (IPR) study (required where mandated by the method).
- Analysis of 4 consecutive Laboratory Control Samples, with acceptable accuracy and precision.
- Where spiking is not possible but QC standards are used ("non-spiked" Laboratory Control Samples), analysis of 4 consecutive Laboratory Control Samples with acceptable accuracy and precision.
- Where one of the three above is not possible, special requirements are as follows:
    - Total Settleable Solids: Successful single-blind PT sample analysis and duplicate results with RPD<10%.
    - Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
    - Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

A flowchart identifying the Demonstration of Proficiency requirements is given in Figure 17-1. The flowchart identifies allowed approaches to assessing Demonstration of Capability when a 4-replicate study is not mandated by the method, when spiking is not an option, or when QC samples are not readily available.

17.2 Continuing Demonstration of Proficiency

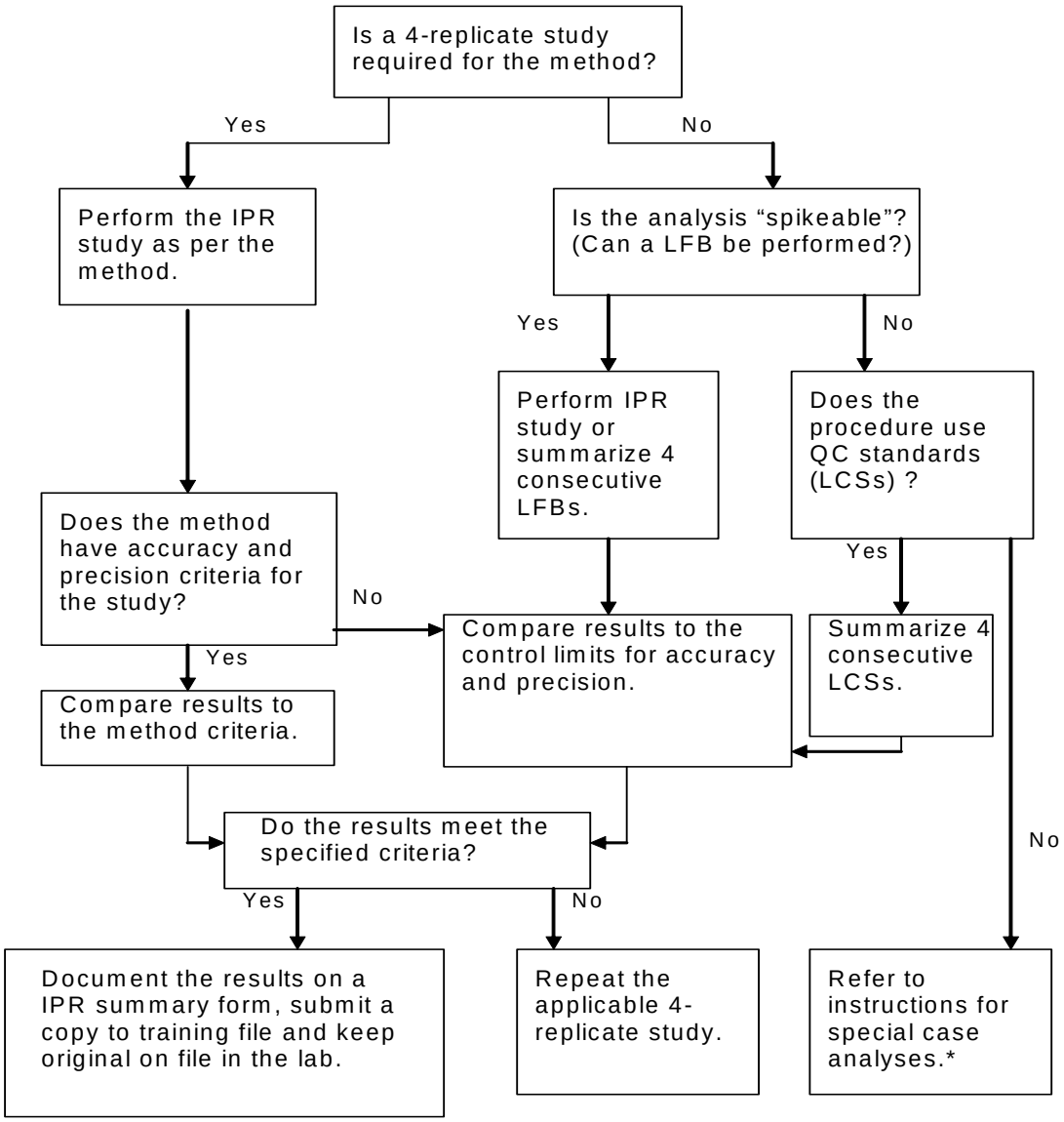
A periodic demonstration of proficiency is required to maintain continuing qualification. Continuing Demonstration of Proficiency is required each year, and may be performed one of the following ways:

- Successful performance on external (independent) single-blind sample analyses using the test method, or a similar test method using the same technology. I.e. PT sample or QC sample blind to the analyst.
- Performing Initial Demonstration of Capability as described above, with acceptable levels of precision and accuracy.
- Analysis of at least 4 consecutive LCSs with acceptable levels of accuracy and precision from in-control analytical batches.
- If the above cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.
- For methods for which PT samples are not available and a spiked analysis (LFB, MDL, etc.) is not possible, analysis of field samples that have been analyzed by another analyst with statistically indistinguishable results.

17.3 Documentation of Training

Records are maintained to indicate the employee has the necessary training, education, and experience to perform their functions. Information of previously acquired skills and abilities for a new employee is maintained in Human Resources personnel files and Columbia Analytical resumes. QA maintains a database to record the various technical skills and training acquired while employed by Columbia Analytical. Information includes the employee's name, a description of the skill including the appropriate method and SOP reference, the mechanism used to document proficiency, and the date the training was completed. General procedures for documenting technical training are described in the *SOP for Documentation of Training (SOP No. ADM-TRANDOC).*

Figure 17-1
Initial Demonstration of Capability Requirements[a]



[a] For IDOC IPR or LFB studies, "second-source" reference materials are used, as per NELAP requirements
*Total Settleable Solids: Successful PT sample analysis and duplicate results with RPD<10%.
*Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
* Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

18.0 REFERENCES FOR ANALYTICAL PROCEDURES – EXTERNAL DOCUMENTS

The analytical methods used at Columbia Analytical generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Typical methods used at Columbia Analytical are taken from the following references:

- National Environmental Laboratory Accreditation Program (NELAP), 2003 Quality Standards.
- American National Standard *General requirements for the competence of testing and calibration laboratories,* ANSI/ISO/IEC 17025:2005(E)
- *Department of Defense Quality Systems Manual for Environmental Laboratories*, Final Version 3 (January 2006).
- *DoD Quality Systems Manual for Environmental Laboratories,* Version 4.1, 4/22/2009
- *Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185 (August 1995).
- *Manual for the Certification of Laboratories Analyzing Drinking Water*, 4th Edition, EPA 815-B-97-001 (March 1997).
- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, (September 1986) and Updates I (July 1992), II (September 1994), IIA (August 1993), IIB (January 1995), III (December 1996), Final Update IV (February 2007), and updates posted online at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm. See Chapters 1, 2, 3, and 4.
- *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, (Revised March 1983).
- *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA/600/R-93/100 (August 1993).
- *Methods for the Determination of Metals in Environmental Samples*, EPA/600/4-91/010 (June 1991) and Supplements.
- *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, EPA 600/4-82-057 (July 1982) and 40 CFR Part 136, Appendix A.
- *Methods for the Determination of Organic Compounds in Drinking Water*, EPA/600/4-88/039 (December 1988) and Supplements.
- *Standard Methods for the Examination of Water and Wastewater*, 18th Edition (1992); 19th Edition (1995), 20th Edition (1998). See Introduction in Part 1000.
- 40 CFR Part 136, Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act.
- 40 CFR Part 141, National Primary Drinking Water Regulations.

- *Analytical Methods for Petroleum Hydrocarbons*, ECY 97-602, Washington State Department of Ecology, June 1997.
- State-specific total petroleum hydrocarbon methods for the analysis of samples for gasoline, diesel, and other petroleum hydrocarbon products (Alaska, Arizona, California, Oregon, Washington, Wisconsin, etc.).
- Annual Book of ASTM Standards, Part 31, Water.
- EPA Contract Laboratory Program, Statement of Work for Organic Analysis, SOW Nos. OLM03.1, OLM03.2, OLM04.2, and OLM04.3.
- EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, SOW No. ILM04.0, ILM04.1, and ILM05.2.
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, EPA-540/R-94/012 (February 1993).
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, EPA-540/R-94/013 (February 1994).
- National Institute for Occupational Safety and Health (NIOSH) *Manual of Analytical Methods*, Third Edition (August 1987); Fourth Edition (August 1994).
- *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*, for USEPA and USACE (March 1986), with revisions through April 1997.
- WDOE 83-13, *Chemical Testing Methods for Complying with the State of Washington Dangerous Waste Regulations* (March 1982) and as Revised (July 1983 and April 1991).
- *Identification and Listing of Hazardous Waste,* California Code of Regulations, Title 22, Division 4.5, Chapter 11.
- *Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Wastewater*, EPA 821-R-93-017 (October 1993).
- *Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters*, EPA 821-B-98-016 (July 1998).
- National Council of the Pulp and Paper Industry for Air and Stream Improvement (NCASI).

APPENDIX A

LIST of QA PROGRAM DOCUMENTS

and

STANDARD OPERATING PROCEDURES

QA Program Files

Quality Assurance Manual	10/2/2009
Software Quality Assurance Plan	7/11/05
CAS-Kelso Certifications/Accreditations	Cert_kel.xls
Columbia Analytical Services MDL Tracking Spreadsheet	Mdl_list.xls
Technical Training Summary Database	TrainDat.mdb
Approved Signatories List	AppSignatories.pdf
Personnel resumes/qualifications	HR Department
Personnel Job Descriptions	HR Department
Quality Control Acceptance Criteria	Qclimits.xls
Master Logbook of Laboratory Logbooks	Masterlog-001
Standard Operating Procedure Database	TrainDat.mdb

Corporate – Policies

POLICY TITLE	POLICY DATE	DATE APPROVED	DATE EFFECTIVE
CAS Quality and Ethics Policy Statement	March 2009	3/19/09	3/19/09
Policy for Data Review and Validation	May 2009	5/5/09	7/1/09
Policy for Internal Quality Assurance Audits	May 2009	5/5/09	7/1/09
Policy for Standards and Reagents Expiration Dates	September 2009	Final draft	9/28/09
Policy for Quality Assurance for Non-Regulated Testing	Draft	-	-
Policy for Use of Accreditation Organization's Name, Symbols, and Logos	Draft	-	-
Policy for Conducting Research, Technical Investigations, and Method Development	In development	-	-

Administrative SOP Corporate

SOP TITLE	SOP Code	Rev	SOP Date
SOP for Checking New Lots of Chemicals for Contamination	ADM-CTMN	4	1/26/09
SOP for Control Limits	ADM-CTRL_LIM	6	9/28/07
SOP for Corrective Action	ADM-CA	5	9/12/07
SOP for Data Recall	ADM-DATARECALL	0	9/21/07
SOP for Document Control	ADM-DOC_CTRL	7	1/27/09
SOP for Documentation of Training	ADM-TRANDOC	10	12/6/07
SOP for Estimation of Uncertainty of Measurements	ADM-UNCERT	4	12/30/08
SOP for Handling Customer Feedback	ADM-FDBK	4	12/10/07
SOP for Making Entries into Logbooks and onto Benchsheets	ADM-DATANTRY	8	9/8/09
SOP for Managerial Review of the Laboratory's Quality Systems	ADM-MGMTRVW	2	11/7/07
SOP for Manual Integration of Chromatographic Peaks	ADM-INT	3	8/28/07
SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation	ADM-MDL	9	9/8/09
SOP for Preparation of Electronic-data for Organic Analyses for Electronic-data Audits	ADM-E_DATA	3	8/29/07
SOP for Preparation of SOPs	ADM-SOP	8	11/14/08
SOP for Preventive Action	ADM-PA	0	11/14/08
SOP for Proficiency Testing Sample Analysis	ADM-PTS	1	9/28/07
SOP for Purchasing Through SOP Purchasing Agent in Kelso	ADM-PUR	2	12/10/07
SOP for Qualification of Subcontract Laboratories Outside of SOP Network	ADM_SUBLAB	4	12/29/08
SOP for Significant Figures	ADM-SIGFIG	8	1/28/09

Administrative SOP Kelso

SOP Title	FILE NAME
CHECKING PIPETTE CALIBRATION	**ADM-CPIP**
CONTINGENCY PLAN FOR LABORATORY EQUIPMENT FAILURE	**ADM-ECP**
CONTROL CHARTING QUALITY CONTROL DATA	**ADM-CHRT**
DATA ARCHIVING	**ADM-ARCH**
DATA REPORTING AND REPORT GENERATION	**ADM-RG**
DEPARTMENT OF DEFENSE PROJECTS LABORATORY PRACTICES AND PROJECT MANAGEMENT	**ADM-DOD**
ELECTRONIC DATA BACKUP AND ARCHIVING	**ADM-EBACKUP**
INTERNAL QUALITY ASSURANCE AUDITS	**ADM-IAUD**
LABORATORY BALANCE MONITORING AND CALIBRATION	**ADM-BAL**
LABORATORY DATA REVIEW PROCESS	**ADM-DREV**
PROJECT MANAGEMENT	**ADM-PCM**
REAGENT LOGIN AND TRACKING	**ADM-RLT**
SUPPORT EQUIPMENT MONITORING AND CALIBRATION	**ADM-SEMC**
SAMPLE BATCHES	**ADM-BATCH**
SAMPLE MANAGEMENT SOPS	**FILE NAME**
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT
SAMPLE DISPOSAL	SMO-SDIS
SAMPLE RECEIVING	SMO-GEN
SAMPLE TRACKING AND LABORATORY CHAIN OF CUSTODY	SMO-SCOC

Technical SOP Kelso

SOP Title	FILE NAME
COLIFORM, TOTAL (DRINKING WATER)	BIO-9221DW
COLIFORM, FECAL	BIO-9221FC
COLIFORM, TOTAL	BIO-9221TC
COLIFORM, FECAL (MEMBRANE FILTER PROCEDURE)	BIO-9222D
COLILERT® and COLITAG	BIO-9223
FECAL STREPTOCOCCUS/ENTEROCOCCUS	BIO-9230B
COLILERT® COMPLETED TEST VERIFICATION OF E. COLI IN MUG CULTURES	BIO-CCT
ENTEROLERT	BIO-ENT
HEPTEROTROPHIC PLATE COUNT	BIO-HPC
MICROBIOLOGY QUALITY ASSURANCE AND QUALITY CONTROL	BIO-QAQC
SHEEN SCREEN/OIL DEGRADING MICROORGANISMS	BIO-SHEEN
EPA CLP ORGANICS ANALYSES	CLP_ORGA
SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION	EXT-3510
CONTINUOUS LIQUID - LIQUID EXTRACTION	EXT-3520
SOLID PHASE EXTRACTION	EXT-3535
SOXHLET EXTRACTION	EXT-3540
AUTOMATED SOXHLET EXTRACTION	EXT-3541
ULTRASONIC EXTRACTION	EXT-3550
WASTE DILUTION EXTRACTION	EXT-3580
SILICA GEL CLEANUP	EXT-3630
REMOVAL OF SULFUR USING COPPER	EXT-3660
REMOVAL OF SULFUR USING MERCURY	EXT-3660M
SULFURIC ACID CLEANUP	EXT-3665
CARBON CLEANUP	EXT-CARCU
DIAZOMETHANE PREPARATION	EXT-DIAZ
FLORISIL CLEANUP	EXT-FLOR
ORGANIC EXTRACTIONS GLASSWARE CLEANING	EXT-GC
PREPARATION OF REAGENTS AND BLANK MATRICES USED IN SEMIVOLATILE ORGANICS ANALYSIS	EXT-REAG
ADDITION OF SPIKES AND SURROGATES	EXT-SAS
SOLID PHASE DISPERSION IN TISSUES	EXT-SPD
MEASURING SAMPLE WEIGHTS AND VOLUMES FOR ORGANIC ANALYSIS	EXT-WVOL
FACILITY AND LABORATORY CLEANING	FAC-CLEAN
OPERATION AND MAINTENANCE OF LABORATORY REAGENT WATER SYSTEMS	FAC-WATER
FLASHPOINT DETERMINATION - SETAFLASH	GEN-1020
COLOR	GEN-110.2
HARDNESS, TOTAL	GEN-130.2
SOLIDS, TOTAL DISSOLVED (TDS)	GEN-160.1
SOLIDS, TOTAL SUSPENDED (TSS)	GEN-160.2
TOTAL SOLIDS	GEN-160.3
SOLIDS, TOTAL VOLATILE AND PERCENT ASH IN SOIL AND SOLID SAMPLES	GEN-160.4
SETTEABLE SOLIDS	GEN-160.5
HALIDES, ADSORBABLE ORGANIC (AOX)	GEN-1650
DETERMINATION OF INORGANIC ANIONS IN DRINKING WATER BY ION CHROMATOGRAPHY	GEN-300.1
ACIDITY	GEN-305.2
ALKALINITY TOTAL	GEN-310.1

PERCHLORATE BY ION CHROMATOGRAPHY	GEN-314.0
CHLORIDE (TITRIMETRIC, MERCURIC NITRATE)	GEN-325.3
CHLORINE, TOTAL/FREE RESIDUAL	GEN-330.4
TOTAL RESIDUAL CHLORINE - METHOD 330.5	GEN-330.5
TOTAL CYANIDES AND CYANIDES AMENABLE TO CHLORINATION	GEN-335
AMMONIA BY FLOW INJECTION ANALYSIS	GEN-350.1
AMMONIA AS NITROGEN BY ION SPECIFIC ELECTRODE	GEN-350.3
NITRATE/NITRITE, NITRITE BY FLOW INJECTION ANALYSIS	GEN-353.2
NITRITE BY COLORIMETRIC PROCEDURE	GEN-354.1
PHOSPHORUS DETERMINATION USING COLORMETRIC PROCEDURE	GEN-365.3
DISSOLVED SILICA	GEN-370.1
GRAVIMETRIC SULFATE	GEN-375.3
SULFIDE, TITRIMETRIC (IODINE)	GEN-376-1
SULFIDE, METHYLENE BLUE	GEN-376-2
PHENOLICS, TOTAL	GEN-420.1
MBAS	GEN-425.1
HALOGENS TOTAL AS CHLORIDE BY BOMB COMBUSTION	GEN-5050
BIOCHEMICAL OXYGEN DEMAND	GEN-5210B
HALIDES, ADSORBABLE ORGANIC (AOX) - SM 5320B	GEN-5320B
TANNIN AND LIGNIN	GEN-5550
CYANIDE EXTRACTION OF SOLIDS AND OILS	GEN-9013
HALIDES, TOTAL ORGANIC (TOX)	GEN-9020
HALIDES, EXTRACTABLE ORGANIC (EOX)	GEN-9020M
TOTAL SULFIDES BY METHYLENE BLUE DETERMINATION	GEN-9030
TOTAL HALIDES BY OXIDATIVE COMBUSTION AND MICROCOULOMETRY	GEN-9076
CARBON, TOTAL ORGANIC IN SOIL	GEN-ASTM
AUTOFLUFF	GEN-AUTOFLU
SULFIDES, ACIDS VOLATILE	GEN-AVS
HEAT OF COMBUSTION	GEN-BTU
CYANIDE, WEAK ACID DISSOCIABLE	GEN-CNWAD
CHEMICAL OXYGEN DEMAND	GEN-COD
CONDUCTIVITY IN WATER AND WASTES	GEN-COND
CORROSIVITY TOWARDS STEEL	GEN-CORR
HEXAVALENT CHROMIUM - COLORIMETRIC	GEN-CR6
CARBONATE (CO3) BY EVOLUTION AND COLUMETRIC TITRATION	GEN-D513-82M
SULFIDE, SOLUBLE DETERMINATION OF SOLUBLE SULFIDE IN SEDIMENT	GEN-DIS.S2
BULK DENSITY OF SOLID WASTE FRACTIONS	GEN-E1109
FERROUS IRON IN WATER	GEN-FeII
FLUORIDE BY ION SELECTIVE ELECTRODE	GEN-FISE
FORMALDEHYDE COLORIMETRIC DETERMINATION	GEN-FORM
HYDROGEN HALIDES BY ION CHROMATOGTRAPHY (METHOD 26)	GEN-HA26
MERCURY IN COAL SAMPLE PREPARATION BY PARR BOMB COMBUSTION	GEN-HGPREP
HYDAZINE IN WATER USING COLORIMETRIC PROCEDURE	GEN-HYD
TOTAL SULFUR FOR ION CHROMATOGRAPHY	GEN-ICS
ION CHROMATOGRAPHY	GEN-IONC
COLOR, NCASI	GEN-NCAS
OXYGEN CONSUMPTION RATE	GEN-O2RATE
CARBON, TOTAL ORGANIC DETERMINATION (WALKELY BLACK METHOD)	GEN-OSU
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Ph IN WATER	GEN-Phw
PARTICLE SIZE DETERMINATION - ASTM PROCEDURE	GEN-PSASTM
PARTICLE SIZE DETERMINATION	GEN-PSP
SULFIDES, REACTIVE	GEN-RS
TOTAL SULFIDE BY PSEP	GEN-S2PS
SULFITE	GEN-SO3
SPECIFIC GRAVITY	GEN-SPGRAV
SUBSAMPLING AND COMPOSITING OF SAMPLES	GEN-SUBS
THIOCYANATE	GEN-THIOCN
NITROGEN, TOTAL AND SOLUBLE KJELDAHL	GEN-TKN
POST DIGESTION DETERMINATION OF TOTAL KJELDAHL NITROGEN BY SEMIAUTOMATED COLORIMETRY	GEN-TKNAA
TOTAL ORGANIC CARBON IN WATER	GEN-TOC
TURBIDITY MEASUREMENT	GEN-TURB
ULTIMATE BOD	GEN-UBOD
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Quantitative Determination of Carbamate Pesticides by High Performance Liquid Chromatography/Tandam Mass Spectrometry (HPLC/MS/MS)	LCP-8321
NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY(HPLC)	LCP-8330B
QUANTITATION OF NITROAROMATICS AND NITRAMINES IN WATER, SOIL, AND TISSUE BY LIQUID CHROMATOGRAPHY AND TANDEM MASS SPECTROMETRY (LC-MS/MS)	LCP-LCMS4
NITROGUANIDINE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	LCP-NITG
QUANTITATION OF NITROPHENOLS IN SOLIS BY LIQUID CHROMATOGRAPHYAND TANDEM MASS SPECTORMETRY (LC-MS/MS)	LCP-NITRO
METHYL MERCURY IN SOIL AND SEDIMENT BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630S
METHYL MERCURY IN TISSUE BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630T
METHYL MERCURY IN WATER BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630W
MERCURY IN WATER BY OXIDATION, PURGE&TRAP, AND COLD VAPOR ATOMIC FLUORES. SPECTROMETRY	MET-1631
MERCURY IN WATER	MET-245.1
METALS DIGESTION	MET-3005A
METALS DIGESTION	MET-3010A
METALS DIGESTION	MET-3020A
METALS DIGESTION	MET-3050B
CLOSED VESSEL OIL DIGESTION	MET-3051M
DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 6020)	MET-6020
ARSENIC BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION	MET-7062
METALS DIGESTION	MET-7195
MERCURY IN LIQUID WASTE	MET-7470A
MERCURY IN SOLID OR SEMISOLID WASTE	MET-7471A/B
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BIOACCESSIBILITY OF METALS IN SOIL AND SOLID WASTE	MET-BIOACC
METALS DIGESTION	MET-DIG
FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRIC ANALYSES	MET-FAA
SAMPLE FILTRATION FOR METALS ANALYSIS	MET-FILT
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DETERMINATION OF TRACE METALS BY GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY (GFAA)	MET-GFAA
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DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 200.8)	MET-ICP.MS
MULTIPLE EXTRACTION PROCEDURE	MET-MEP
TRACE METALS IN WATER BY PRECONCENTRATION USING REDUCTIVE PRECIPITATION FOLLOWED BY ICP-MS	MET-RPMS
WASTE EXTRACTION TEST (WET) PROCEDURE (STLC) for NONVOLATILE and SEMIVOLATILE PARAMETERS	MET-STLC
METALS AND SEMIVOLATILES TCLP EXTRACTION (EPA METHOD 1311)	MET-TCLP
SAMPLE PREPARATION OF BIOLOGICAL TISSUES FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS	MET-TDIG
TISSUE SAMPLE PREPARATION	MET-TISP
GRAVIMETRIC DETERMINATION OF HEAXANE EXTRACTABLE MATERIAL (1664)	PET-1664
GASOLINE RANGE ORGANICS BY GAS CHROMATOGRAPHY	PET-GRO
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ANALYSIS OF SOLID AND AQUEOUS SAMPLES FOR STATE OF WISCONSIN DIESEL RANGE ORGANICS	PHC-WIDRO
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT
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CONGENER-SPECIFIC DETERMINATION OF PCBS BY GC/ECD - METHOC 8082A	SOC-8082ACo
PCBS AS AROCLORS	SOC-8082Ar
CONGENER-SPECIFIC DETERMINATION OF PCBS BY GC/ECD	SOC-8082C
DETERMINATION OF NITROGEN OR PHOSPHORUS CONTAINING PESTICIDES	SOC-8141
CHLORINATED HERBICIDES	SOC-8151
CHLORINATED PHENOLS METHOD 8151 MODIFIED	SOC-8151M
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SOC-8270C
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - METHOD 8270D	SOC-8270D
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - LOW LEVEL PROCEDURE	SOC-8270L
POLYNUCLEAR AROMATIC HYDROCARBONS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY SIM	SOC-8270P
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POLYNUCLEAR AROMATIC HYDROCARBONS BY HPLC	SOC-8310
ALDEHYDES BY HPLC	SOC-8315A

NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	SOC-8330
NITROGLYCERIN AND PETN BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	SOC-8332
RESIN AND FATTY ACIDS BY GC/MS - NCASI METHOD 85.02 MODIFIED	SOC-85.02
METHANOL IN PROCESS LIQUIDS AND STATIONARY SOURCE EMISSIONS	SOC-9403
HAZARDOUS AIR POLLUTANTS (HAPS) IN PULP AND PAPER INDUSTRY CONDENSATES	SOC-9901
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BUTYLTINS	SOC-BUTYL
CALIBRATION OF INSTRUMENTS FOR ORGANICS CHROMATOGRAPHIC ANALYSES	SOC-CAL
CALIBRATION OF INSTRUMENTS FOR ORGANICS CHROMATOGRAPHIC ANALYSES USING EPA 8000C	SOC-CAL8000C
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CPSC PHTHALATES BY GC/MS SELECTIVE ION MONITORING	SOC-CPSC
DIMP	SOC-DIMP
DMD SYNTHESIS	SOC-DMD
TOTAL OLEANOLIC ACID SAPONINS IN WATER BY ACID HYDROLYSIS AND HPLC/MS/MS	SOC-LCMS3
PERCENT LIPIDS IN TISSUE	SOC-LIPID
MONOCHLOROACETIC ACID BY GC-ECD	SOC-MCA
NONYLPHENOLS ISOMERS AND NONYLPHENOL ETHOXYLATES	SOC-NONYL
ORGANIC ACIDS IN AQUEOUS MATRICES BY HPLC	SOC-OALC
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CHLORINATED PESTICIDES BY GC/MS/MS, EPA METHOD 1699 MODIFIED	SOC-PESTMS2
PERFLUORINATED COMPOUNDS BY HPLC/MS/MS	SOC-PFC
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POLYBROMINATED DIPHENYL ETHERS (PBDEs) AND POLYBROMINATED BIPHENYLS (PBBs) BY GC/MS	SOC-ROHS
SEMI-VOLATILE ORGANICS SCREENING	SOC-SCR
1,2-DIBROMOETHANE, 1,2-DIBROMO-3-CHLOROPROPANE, AND 1,2,3-TCP BY GC	SVD-504
ORGANOCHLORINE PESTICIDES AND PCBS IN DRINKING WATER	SVD-508_1
CHLORINATED HEBICIDES IN DRINKING WATER	SVD-515_4
N-NITROSAMINES BY GC/MS/MS	SVD-521
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS (METHOD 525.2)	SVD-525
SELECTED PESTICIDES AND FLAME RETARDANTS IN DRINKING WATER BY GC/MS (EPA METHOD 527)	SVD-527
DETERMINATION OF EXPLOSIVES AND RELATED COMPOUNDS IN DRINKING WATER BY GC/MS	SVD-529
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AROMATIC VOLATILE ORGANICS (BTEX) BY GC - METHOD 602	VOC-602BTEX
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-624
AROMATIC VOLATILE ORGANICS (BTEX) BY GC - METHOD 8021	VOC-8021BTEX
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VOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTIVE ION MONITORING	VOC-8260S

VOA STORAGE BLANKS	VOC-BLAN
SAMPLE SCREENING FOR VOLATILE ORGANIC COMPOUNDS IN SOIL, WATER AND MISC. MATRICES	VOC-BVOC
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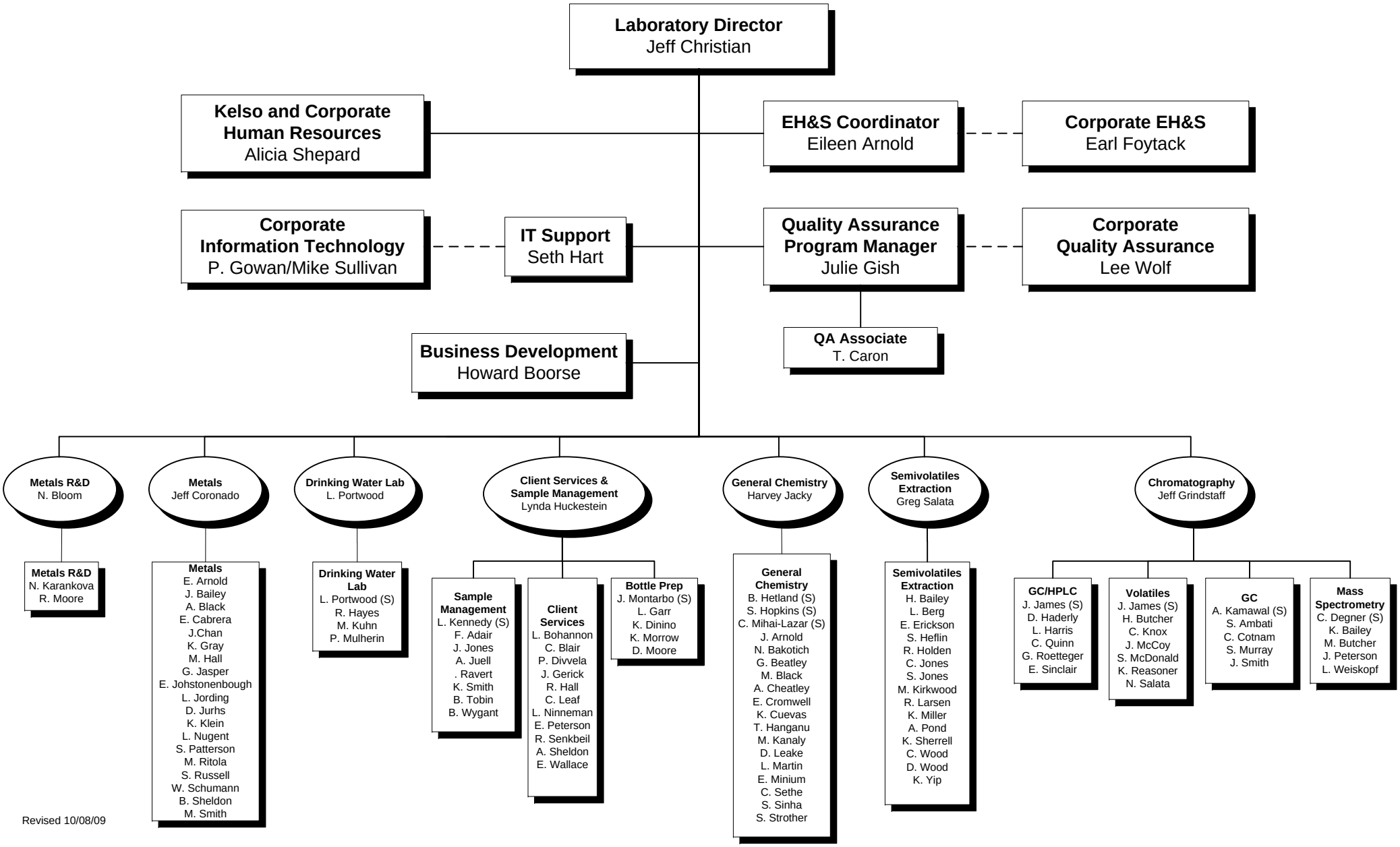
APPENDIX B

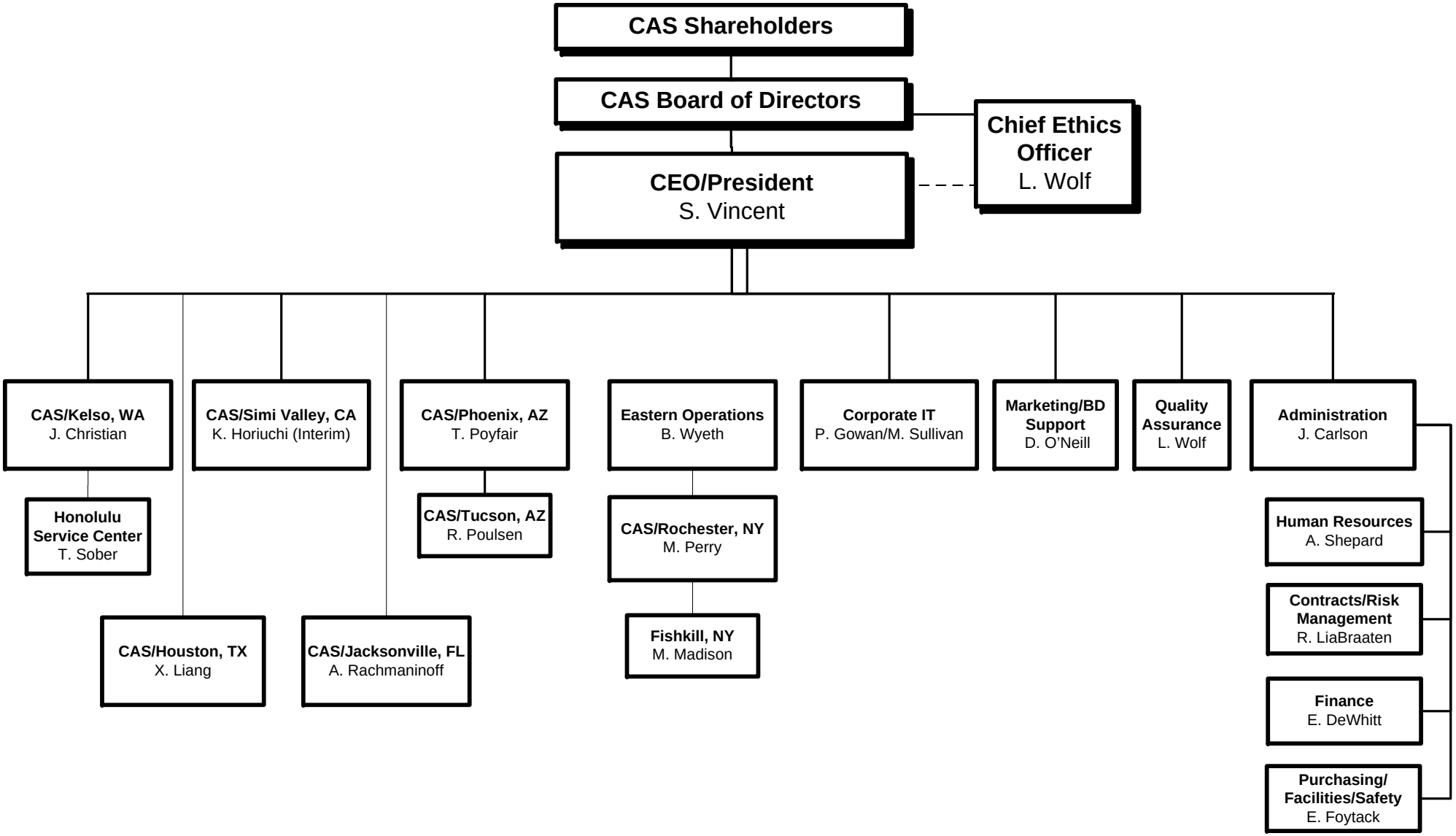
ORGANIZATIONAL CHARTS and RESUMES OF KEY PERSONNEL

Environmental and General Testing Division
Kelso, Washington
Laboratory Organization



Revised 10/08/09

Laboratory Division Organization



Revised .12/1/09

JEFFREY D. CHRISTIAN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**VICE PRESIDENT/NW REGIONAL DIRECTOR –** 1996 to Present
Responsibilities	Responsible for all phases of laboratory operations at the Kelso (WA) facility, including project planning, budgeting, and quality assurance. Primary duties include the direct management of the Kelso laboratory (i.e. serves as the Kelso Laboratory Director, 1993-present). Also responsible for additional duties acquired as a member of the Columbia Analytical Services Holdings, Inc., Board of Directors.
Experience	**Laboratory Director, Kelso Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1993-1995. Responsible for all phases of laboratory operations, including project planning, budgeting, and quality assurance. **Operations Manager, Kelso Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1993. Responsibilities included directing the daily operation of the Kelso laboratory. Other responsibilities and duties included functioning as a technical consultant to clients, providing assistance in developing and planning analytical schemes to match client objectives, and writing and developing analytical procedures/methods. Also, served as Project Manager for State of Alaska Department of Environmental Conservation contract and Coordinator for EPA Special Analytical Services (SAS) contracts. **Project Chemist and Manager, Metals Analysis Laboratory,** *Columbia Analytical Services, Kelso, Washington,* 1989-1992. Responsible for directing the daily operation of the Metals Laboratory, including the sample preparation, AAS, ICP-OES, and ICP-MS Laboratories. **Scientist,** *Weyerhaeuser Technology Center, Federal Way, Washington,* 1986-1989. Responsibilities included supervising atomic spectroscopy laboratory which included flame and furnace AAS, ICP-OES, and sample preparation capabilities to handle a wide variety of sample types. Interfaced with internal and external clients to provide technical support. Wrote and developed analytical procedures/methods. **Lead Technician, Metals Lab,** *Weyerhaeuser Technology Center, Federal Way, Washington*, 1981-1986. Responsibilities included primary ICP and AAS analyst for EPA-CLP contract work. Extensive experience in wide variety of environmental and product-related testing. **Research Assistant,** *ITT Rayonier, Olympic Research Division, Shelton, Washington,* 1978-1981. Responsibilities included performing water quality tests, product-related analytical tests, corrosion tests (i.e., potentiometric polarization techniques), and operated pilot equipment specific to the pulp and paper industry.
Education	**B.S., Chemistry,** *Evergreen State College, Olympia, Washington,* 1993. **ICP/MS Training Course,** *VG-Elemental,* 1992. **Coursework,** *Pacific Lutheran University, Tacoma, Washington.* 1988-1989. **Coursework,** *Tacoma Community College, Tacoma, Washington.* 1970-1971, 1988-1989. **Perkin-Elmer Advanced Furnace,** *Norwalk, Connecticut,* 1986. **CERTIFICATION, Chemistry,** *L.H. Bates Technical, Tacoma, Washington,* 1978. **Coursework,** *Central Washington University, Ellensburg, Washington.* 1969-1970.
Publications/ Presentations	*Mr. Christian has a number of publications and presentations. For a list of these publications and presentations, please contact CAS.*

JULIE GISH

1996 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**TECHNICAL MANAGER I, KELSO LAB QUALITY ASSURANCE MANAGER** – 2008 to Present
Responsibilities	Responsible for the overall implementation of the laboratory QA program. Responsible for the Quality Assurance Manual, certifications, documenting SOPs, and maintaining proficiency testing (PT) records. Oversee balance calibration and sample storage temperature control. Maintain certifications/accreditations for regulatory agencies and client certifications or approval programs. Act as primary point of contact during laboratory audits and provides audit responses and initiates any corrective actions. Coordinate the analysis and reporting of PT samples. Conduct internal audits and make recommendations for corrective action.
Experience	**Scientist IV, Semi-Volatile Mass Spectrometry Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 2002-2008. Primary responsibilities were analysis, interpretation and report generation for semivolatile organics by GC/MS. Analyses included EPA 625, 8270, SIM, and other miscellaneous methodology.
	Technical Manager I, Semi-Volatile GC Organics Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1999-2002. Primary responsibilities include supervision and oversight of semi-volatile GC department. This includes initiating new methods, staff training, workload management, and instrument maintenance/troubleshooting. Duties include departmental compliance with CAS QA and Safety policies. Responsible for analysis, interpretation and report generation for pesticides and PCB's by EPA Methods 608, 8080, 8081, 8082, EPA 8141A, Organotins, and CLP Pesticides.
	Scientist III, Semi-Volatile Organics Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1996-1999. Primary responsibilities were analysis, interpretation and report generation for pesticides and PCB's by EPA Methods 608, 8080, 8081, 8082, and CLP-Pesticides. Secondary responsibilities include organics semi-volatile sample preparation.
	Scientist, Volatile Organics Sample Preparation, *Employer's Overload, Longview, Washington – assigned to the Columbia Analytical Services, Inc., Kelso, Washington facility,* 1996. Primary duties included the preparation of water, soil, sediment and tissue samples using EPA Methods 3510, 3520, 3540, 3550, and 3545. Other duties were the further clean up of extracts using EPA Methods 3620 (Florsil), 3610 (Alumina), 3630 (Silica gel), 3650 (Acid/Base Partitioning), and 3660 (Sulfur).
	Organics Chemist and GC/MS Chemist, *Coffey Laboratories, Portland, Oregon,* 1990-1996. Primary responsibilities included sample preparation and analysis for EPA FID, ECD, and HPLC using various EPA SW-846 and 500-series methods, as well as other methodology. Later, moved to GC/MS position which included sample preparation, analysis, and associated instrument maintenance for EPA Methods 625, 8027, and 525 BNA's. Also responsible for data review and approval of data packages.
	QC Manager/QC Supervisor and Product Manager, Corn Products, *Frito-Lay, Inc., Vancouver, Washington,* 1982-1990. Manager of the QC department overseeing three supervisors and approximately 30 technicians. Responsible for department cost, accuracy, timeliness of data and safety performance. Later, responsible for production oversight of brand name snacks. Responsible for cost, quality and safety performance over three shifts. Managed four supervisors directly and approximately 60 employees indirectly.
	Food Technologist, QA Department, *Kraft, Inc., Buena Park, California*, 1978-1981. Responsible for audits, formulations, finished product evaluation, batch reviews and technical support.
Education	**MS, Food Science, Minor in Industrial Engineering,** *Oregon State Univ. Corvallis, Oregon,* 1978. **BS, Food Science, Minor in Business Administration,** *Utah State University, Logan, Utah,* 1975
Publications/ Presentations	*Quality Improvement Team Leader,* Coffey Laboratories, Portland, Oregon. 1991
	Methods Improvement Program, Coffey Laboratories, Portland, Oregon. Seminars on Development and Implementation 1990.
	Statistical Process Control and Total Quality Management, Frito-Lay, Vancouver, Washington. Routine Training Classes 1986-1988.

GREGORY G. SALATA

2003 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**PROJECT/EXTRACTIONS MANAGER V** – 2003 to Present
Responsibilities	Responsibilities include Project Management, including quotation preparation and data reporting, as well as providing technical support to the laboratory as needed. Responsibilities also include oversight of the organic extractions lab, managing resources and providing technical support for all organic preparation work flows. 2003-Present.
Experience	**Project Manager,** *B&B Laboratories, College Station, Texas,* 1999-2003. Supervisor/responsible for analysis of TPH (waters, tissues, sediments), organotins (waters, tissues, sediments), Atterberg Limits (sediments), and total organic/inorganic carbon (sediments, waters). Also responsible for report generation on specific projects. Instrumentation operated included GCs with FID and FPD detectors, Combustion TOC, Water TOC, and Dionex Accelerated Solvent Extractor.
	Graduate Student, *Texas A&M University, College Station, Texas,* 1991-1999. While working toward MS in Oceanography, performed organic extractions for pesticides, PCBs, PAHs, and butyltins. While working toward Ph.D. in Oceanography determined stable carbon isotope ratios in sediments, waters, and bacterial phospholipid fatty acids. Other responsibilities included field sample collection, and operation/maintenance of FinniganMAT 252 isotope ratio MS.
	Analytical Chemist, *Science Applications International (SAIC), San Diego, California,* 1989-1990. Performed organic extraction and GC/FID analysis on sediment/rock samples for the Exxon Valdez oil spill.
	GC Chemist, *Analytical Technologies, San Diego, California,* 1987-1989. Responsible for analysis of volatile organics using purge and trap and GC/PID/ELCD.
Education	**Ph.D., Oceanography,** *Texas A&M University, College Station, Texas.* 1999 **MS, Oceanography,** *Texas A&M University, College Station, Texas.* 1993 **BA, Chemistry,** *University of California San Diego, Revelle College, La Jolla, California.* 1987
Publications/ Presentations	*Dr. Salata has a number of publications and published abstracts. For a list of these publications and published abstracts, please contact CAS.*
Affiliations	Society of Environmental Toxicology and Chemistry (SETAC)
	American Chemical Society

JEFFREY A. CORONADO

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**TECHNICAL MANAGER IV, INORGANICS DEPARTMENT MANAGER –** 2001 to Present
Responsibilities	Oversee the operation of the Metals Group. Responsible for the quality and timeliness of the inorganic laboratories analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation. **Documentation of Demonstration of Capabilities is available for review.**
Experience	**Metals Department Manager,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-2001. Responsibilities included management of all aspects of the metal laboratory operation, including personnel training and evaluation, review of all metals data, and report generation. Also responsible for client service on a number of ongoing CAS accounts. Technical duties include primary analytical responsibility for trace level metals analysis by ICP/MS. Analyses range from routine water and soil analysis, to marine tissues, as well as industrial applications such as ultra-trace QA/QC work for various semiconductor clients. Also responsible for a number of specialized sample preparation techniques including trace metals in seawater by reductive precipitation, and arsenic and selenium speciation by ion-exchange chromatography. Developed methodology for performing mercury analysis at low part per trillion levels by cold vapor atomic fluorescence.. **Supervisor, GFAA Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1989-1992. Responsibilities included supervision of metals analysis by graphite furnace atomic absorption following SW-846 and EPA CLP methodologies. Duties include workload scheduling, data review, instrument maintenance, personnel training and evaluation.
Education	**Field Immunoassay Training Course**, *EnSys Inc*., 1995. **Winter Conference on Plasma Spectrochemistry**, *San Diego, California,* 1994. **ICP-MS Training Course**, *VG-Elemental*, 1992. **BS, Chemistry,** *Western Washington University, Bellingham, Washington,* 1988. **BA, Business Administration,** *Western Washington University, Bellingham, Washington,* 1985.

LYNDA A. HUCKESTEIN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**CLIENT SERVICES MANAGER IV** – 1998 to Present
Responsibilities	Management of the Client Services Departments: Project Management, Electronic Data Deliverables and Report Generation, and Sample Management. Personally responsible for approximately 1.5 million dollars of client work annually performing technical project management and client service. Provides technical and regulatory interpretation assistance, as well as project organization of work received by the laboratory.
	Documentation of Demonstration of Capabilities is available for review.
Experience	**Project Chemist,** *Columbia Analytical Service, Inc., Kelso, Washington,* 1992-1998. Primary responsibilities included technical project management and client service in areas of pulp & paper, marine services, mining, and DOD. Also responsible for providing technical and regulatory interpretation assistance as-well-as project organization to work received by the laboratory
	Project Chemist and Department Manager, General Chemistry Laboratory, *Columbia Analytical Services, Inc.,* 1989-1992. Responsible for management of the General Chemistry laboratory for routine wastewater, bioassay, and microbiological analyses. Also responsible for supervision of staff, data review, and reporting.
	Analyst III, *Columbia Analytical Services, Inc., Kelso, Washington*, 1989. Primary responsibilities included coliform testing, total recoverable petroleum hydrocarbon extractions and analysis, BODs, ammonias, and TKN, in addition to miscellaneous wet chemistry analyses.
	Microbiologist/Chemist, *Coffey Laboratories, Portland, Oregon,* 1983. Coliform analysis; water chemistry.
	Laboratory Assistant, *Oregon State University, Corvallis, Oregon,* 1983. Wheat spike dissection and tissue culture.
Education	**BS, Microbiology,** *Oregon State University, Corvallis, Oregon,* 1983.

HARVEY L. JACKY

1999TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**TECHNICAL MANAGER II** – 2008 to Present
Responsibilities	Oversee the operation of the General Chemistry and Microbiology groups. Responsible for the quality and timeliness of the inorganic laboratories analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation. **Documentation of Demonstration of Capabilities is available for review.**
Experience	**Project Manager III,** *Columbia Analytical Services, Inc., Kelso, WA*, 1999-2008. Responsible for technical project management, ensuring overall data quality and compliance with customer requirements, and providing technical support to clients regarding laboratory application to projects. Additionally, acts as a consultant to clients regarding industrial/environmental compliance issues; serving as liaison between clients and regulatory agencies. **Director of Project Management,** *Coffey Laboratories, Portland, Oregon*, 1997-1999. Responsible for technical project management. Communicated with clients to determine needs and expectations. Monitored laboratory production and ensured the timely completion of analytical projects. Technical consultant for clients regarding environmental compliance. Supervised and managed other members of the project management team. Served as a member of the senior management team for oversight of general operations, strategic planning, finances, and policy. **Project Manager/Chemist,** *Coffey Laboratories, Portland, Oregon*, 1997-1999. Served as primary liaison between Coffey Laboratories and major clients. Ensured that work was completed in a timely manner and done to client specifications. Served as technical consultant regarding environmental chemistry, soil remediation, and waste water industrial compliance. Clients included the Oregon Department of Transportation, Hazmat Unit, Portland, Oregon; Raythion Demilitarization Co., Umatilla, Oregon; Hydroblast - Wastewater Evaporator Systems, Vancouver, Washington; and Union Pacific Railroad, Northwest Region, Klamath Falls, Oregon. **Technical Sales Representative,** *Coffey Laboratories, Portland, Oregon*, 1995-1997. Responsible for marketing and sales, including actively prospecting for new potential clients. Additional responsibilities included procurement and preparation of all major project bids; ensuring that client expectations were met; and maintaining customer satisfaction. Served as consultant regarding industrial compliance issues, environmental remediation projects, and hazardous waste management. **Senior Chemist/Laboratory Chemical Hygiene Officer,** *Coffey Laboratories, Portland, Oregon*, 1988-1995. Performed analytical tests including Anions by Ion Chromatography (EPA 300.0), PAHs by HPLC (EPA 8310), Cyanides (EPA 335), and other inorganic, wet chemistry, and organic analytical tests on a wide variety of sample matrices. Responsible for the initial quality assurance review of work performed, supervised and managed personnel. Developed and implemented Laboratory Chemical Hygiene Plan. Directed personnel in regards to safety issues and hazardous waste management. Served as consultant and teacher regarding analytical methodology, environmental compliance, and industrial hygiene.
Education	**40-Hour Hazmat Certification,** *PBS Environmental,* 1996. **Industrial Emergency Response,** *SFSP Seminar,* 1991 **BS, Zoology,** *Oregon State University, Corvallis, Oregon,* 1988. **BS, General Science,** *Oregon State University, Corvallis, Oregon,* 1988. **COURSEWORK, General Studies,** *Linfield College, McMinnville, Oregon,* 1981-1982.
Publications/ Presentations	*Biochemical and Physical Factors Involved in the Application and Measurement of a Soil Bioremediation System.* Biogeochemistry, Portland State University, 1996
Affiliations	American Chemical Society, Member since 1988

JEFFERY A. GRINDSTAFF

1991 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**TECHNICAL MANAGER III, PHARMACEUTICAL, GC/MS VOA AND SEMI-VOA LABORATORIES, –** 1997 to Present
Responsibilities	Primary responsibilities include leadership of the Pharmaceutical, GC/MS VOA and Semi-VOA staff, management of method development, training, data review, tracking department workload, scheduling analyses. Responsible for ensuring data quality and timeliness. Also responsible for project management and coordination for pharmaceutical clients.
	Documentation of Demonstration of Capabilities is available for review.
Experience	**Manager, GC/MS VOA Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1994-1997. Responsible for supervision of GC/MS VOA staff, method development, training, data review, tracking department workload, scheduling analyses, and general maintenance and troubleshooting of GC/MS systems.
	Scientist III, GC/MS VOA Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1991-1994. Responsibilities included scheduling workload, data review, instrument maintenance and troubleshooting, and personnel training and evaluation. Also responsible for supervision of extraction personnel and instrument analysts. Additional supervisory duties included report generation and data review for GC analyses. Responsibilities also included project management and customer service.
	Chemist, *Enseco-CRL, Ventura, California,* 1990-1991. Established GC/MS department including inventory maintenance, preparation of state certification data packages, method development, SOPs, and extended data programs. Performed daily maintenance and troubleshooting of GC and GC/MS instrumentation. Scheduled and performed routine and non-routine VOA analyses.
	GC/MS Chemist, VOA Laboratory *Coast-to-Coast Analytical Service, San Luis Obispo, California,* 1990-1991. Responsible for standard preparation for VOA analyses, instrument calibration, tuning, and maintenance. Also implemented and further developed EPA methods for quantitative analysis of pesticides and priority pollutants.
Education	**Sampling and Testing of Raw Materials***, PTI International, 2004*. **Leadership Training,** *Richard Rogers Group, 1996* **Mass Selective Detector Maintenance,** *Hewlett Packard Education Center, 1993* **Interpretation of Mass Spectra I,** *Hewlett-Packard Analytical Education Center,* 1992. **B.S., Chemistry**, *California Polytechnic State University, San Luis Obispo, California,* 1989. **A.A., Liberal Arts,** *Allan Hancock College, Santa Maria, California.* 1986
Publications/ Presentations	*Low Level Analysis of 1,4-Dioxane by GC/MS SIM using Large Volume Injection,* with J. Peterson and R. Holden. SETAC National Meeting Poster Session, Portland, OR 2004.
	Low Level Determination of N-nitrosodimethylamine by Chemical Ionization GC/MS with Large Volume Injection, with C. Degner and J. Peterson. SETAC National Meeting Poster Session, Portland, OR 2004.
	Analysis of Polybrominated Diphenyl Ethers by GC/MS with Large Volume Injection, J. Peterson and M.Thompson SETAC National Meeting Poster Session, Portland, Oregon, 2004.
	Alternate Method to Lower Detection Limits to Satisfy Regulatory Action Levels for Volatiles in Groundwater, with David Edelman, Kairas Parvez, and Paul Laymon. TAPPI National Meeting, Orlando, FL 1996
Affiliations	American Chemical Society. 1989

NICOLAS BLOOM

2008 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position **Scientist VII** – 2008 to Present

Responsibilities **Senior Research Scientist**

Mr. Bloom has been involved in research on the biogeochemistry of trace metals in the environment for 30 years. After graduating from the University of Washington in 1979, he entered the graduate program in the Civil Engineering Department, where he worked as a full time researcher, investigating the sorption behavior of ultra-trace concentrations of cations and anions on ferric hydroxide suspensions. In 1980, Mr. Bloom was hired by the Battelle Marine Research Laboratory to develop sampling and analytical techniques to quantify a wide range of trace metals in sea water at ambient levels and apply those methods to the biogeochemical cycling of Hg, As, Ag, Pb, Cd, and Cu in Puget Sound. In 1984 Mr. Bloom returned to graduate school at the University of Connecticut, where he developed analytical techniques to allow the speciation of Hg at the sub-picogram level by GC-CVAFS. These methods have since been applied to investigate the cycling of Hg and its various compounds in lacustrine and marine systems throughout the world.

In 1991, Mr. Bloom founded Frontier Geosciences Inc., where he continued research into ultra-low level metals speciation in sediments, air, and fossil fuels, as well as mentored the development of IC-ICP/MS and IC-HG-AFS methods for most other trace metals and for Se, As, and Cr speciation. From 2001-2005, Mr. Bloom collaborated extensively with the Universita Ca'Foscari di Venezia in a study of Hg speciation and dynamics in the Venice Lagoon. In 2004, Mr. Bloom founded Studio Geochimica LLC, continuing his studies of the biogeochemistry of trace metals in the environment and industry. In 2008, Mr. Bloom joined Columbia Analytical Services, as Director of the Trace Metals Research and Development Department. In this position, Mr. Bloom is responsible for the development and validation of new trace metals speciation methodologies as well as working with clients and staff having biogeochemical questions or particularly perplexing analytical issues.

Experience

Research Scientist, *Battelle Pacific Northwest Laboratory, Marine Sciences Lab, Sequim, WA,* 1980-1989. As an analyst, developed and validated ultra-clean sampling methods and techniques for the analysis of all 13 EPA priority trace metals in water, sediment, and tissues with detection limits below the ambient background concentrations. As a researcher, emphasized biogeochemical processes of trace metals, particularly at the air/sea and sediment/water interfaces. Supervised two technicians.

Owner/Manager/Sr. Scientist, *Studio Geochimica LLC, Seattle, WA*, 2004 - 2008. Set up the scientific agenda, marketing, sales, inventing new analytical methods, mentoring, working in the lab as scientist and analyst, etc. Staff varied from 4 to 9 people.

Owner/Manager/Sr. Scientist, *Frontier Geosciences Inc., Seattle, WA*, 1991 – 2004. Set up the scientific agenda, marketing, sales, inventing new analytical methods, mentoring, working in the lab as scientist and analyst, etc. Staff varied from 3 in 1991 to 87 people in 2003.

Education

BS, Chemistry, *University of Washington, Seattle, WA*, 1979.

MS, Chemical Oceanography, *University of Connecticut, Storrs, CT* 1986.

Publications/ Presentations

Nicolas Bloom Mr. Bloom has approximately 120 publications on the biogeochemistry and analysis of trace metals in the environment (please inquire for publication list or copies of key papers), and has over 400 presentations at conferences and symposia world-wide.

Affiliations

ASTM, ACS (past member), ASLO (past member)

LOREN E. PORTWOOD

1992 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position

Technical Manager I, DRINKING WATER LABORATORY – 2008 to Present

Responsibilities

Responsible for the overall operation and supervision of the Organic Drinking Water department. Also responsible for implementation and oversight of UCMR2 analyses. Perform method development. Project management of drinking water accounts. Development of Standard Operating Procedures for Drinking Water methods. Operation of Varian GC/MS, Agilent GC/ECD and Agilent HPLC.

Documentation of Demonstration of Capabilities is available for review.

Experience

Scientist IV, Drinking Water Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 2002-2008. Plan, conduct, and, as lead analyst, supervise analyses using advanced instrumentation such as HPLC with post column derivatization, GC/MS, and GC/ECD. Responsible for data interpretation, quality control and data reporting. Additional responsibilities include preparation of SOPs and specifications for processes and tests; handling routine and advanced maintenance and troubleshooting of instrumentation; and assisting in the training of staff department analysts. Assists the department manager and/or other senior scientists in setting up more complex procedures. Serves as senior technical advisor for teams and projects.

Technical Manager I, Petroleum Hydrocarbon Laboratory Supervisor, *Columbia Analytical Services, Inc., Kelso, Washington,* 1998-2002. Primary responsibilities include organizing and prioritizing the workload for the petroleum hydrocarbon team, initiating new methods and process improvements, and staff development and training. Other duties include department wide compliance with CAS quality assurance guidelines, routine system checks, assist and encourage staff in troubleshooting equipment and procedural problems, and lead by example in a manner that is consistent with company, state and federal guidelines. Also responsible for duties listed below under Scientist II and Scientist III.

Scientist III, Petroleum Hydrocarbon Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1997-1998. Duties primarily as listed below.

Scientist II, **Petroleum Hydrocarbon Laboratory,** Columbia Analytical Services, Inc., Kelso, Washington, 1996-1997. Primary responsibilities included analysis, reporting, and archiving of water, soil, and product samples for semi-volatile petroleum hydrocarbons and miscellaneous FID tests. Methods of analysis include EPA methods 8100, 8310, 8315, 8330, 8040, 8015 and various state modifications of 8015 (OR, WA, CA, AK). Additional analyses include solvent scans, alcohols, glycols, and EPA methods 413.2 and 418.1. Other responsibilities include sample preparation and instrument maintenance.

Scientist I, Petroleum Hydrocarbon Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1993-1996. Primary responsibilities included the analysis, reporting, and archiving of water, soil, and product samples for semi-volatile petroleum hydrocarbons. Methods of analysis include EPA method 8015 and various state modifications thereof (OR, WA, CA, AK). Additional responsibilities include sample preparation, instrument maintenance, and assistance with other departmental analyses, including EPA methods 413.2 & 418.1.

Bench Chemist I, Organic Extractions Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1992-1993. Primary responsibilities included the performance of a full range of semi-volatile sample preparations for water, soil, and oil to be analyzed in the GC, GC/MS, and Petroleum Hydrocarbon Laboratories. These extraction methods included hazardous waste, wastewater, and drinking water procedures. Other responsibilities included extract cleanup via Florisil®, GPC, and Hg.

Chemist, Treclen Laboratories, Spokane, Washington, 1990-1992. Primary responsibilities included inorganic water and soil testing by EPA methods. As Chemist, I developed the testing which was accredited by the EPA, which included everything from metal digestions, to phosphates, to TSS and TDS.

Education

Comprehensive HPLC Training, *Restek,* 2002.
Purge & Trap Theory and Troubleshooting, *Full Spectrum Analytics, Inc.,* 2001.
HP5890 GC Advanced Operations, *Hewlett Packard,* 1996.
HP6890 Fast GC, *Hewlett Packard,* 1996.
Quality Training, *Roger Tunks,* 1996.
Capillary Chromatography Training, *Restek,* 1993.
HP5890 GC Maintenance and Troubleshooting, *Hewlett Packard,* 1993.
BS, Chemistry, Emphasis in Biochemistry, *Whitworth College, Spokane, Washington*, 1990.

EILEEN M. ARNOLD

1987 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**SCIENTIST IV, METALS LABORATORY, KELSO HEALTH AND SAFTEY OFFICER –** 1994 to Present
Responsibilities	Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits. **Documentation of Demonstration of Capabilities is available for review.**
Experience	**Project Chemist, Client Services Group, Kelso Health and Safety Officer,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1994. Duties included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to act as liaison for all client-related activities within Columbia Analytical Services, Inc. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits. **Scientist IV, Metals Laboratory, Health and Safety Officer,** *Columbia Analytical Services, Inc., Kelso, Washington, 1987-1992.* Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits. **Chemist,** *Dow Corning Corporation, Springfield, Oregon,* 1986-1987. Responsibilities included ICP and atomic absorption work in silicon manufacturing. Methods development for ICP analysis of minor impurities found in silicon. **Chemist,** *Ametek, Inc., Harleysville, Pennsylvania,* 1982-1985. Responsibilities included product research and development chemist involved in production of thin-film semiconductors for use as solar cells. Work involved AA and SEM techniques. **Chemist,** *Janbridge, Inc., Philadelphia, Pennsylvania*, 1978-1982. Responsibilities included maintaining electroplating process lines through wet chemical analysis techniques, and performed Quality Assurance testing on printed circuit boards.
Education	**BA, Chemistry,** *Immaculata College, Immaculata, Pennsylvania,* 1977.
Affiliations	American Chemical Society, Member since 1987.

APPENDIX C

MAJOR ANALYTICAL EQUIPMENT

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balances (10):			
Precisa and Mettler models	1988-2008	MM	15
Autoclave - Market Forge Sterilmatic	1988	LM	5
Autotitrator – Thermo Orion 500	2007	LM	3
Calorimeters (2):			
Parr 1241 EA Adiabatic	1987	LM	4
Parr 6300 Isoparabolic	2005	LM	4
Centrifuge - Damon/IEC Model K	1992	LM	15
Colony Counter - Quebec Darkfield	1988	LM	4
Conductivity Meters (2):			
YSI Model 3200	2004	LM	4
VWR	2001	LM	4
Digestion Systems (5):			
COD (4)	1987, 1989	LM	5
Kjeldahl, Lachat 46-place (1)	1999	LM	3
Dissolved Oxygen Meter - YSI Model 58 (3)	1987, 1988, 1991	LM	5
Distillation apparatus (Midi) - Easy Still (2)	1996, 2000	LM	7
Drying Ovens (11):			
Shel-Lab and VWR models	1988 - 2003	LM	15
Flash Point Testers (2):			
ERDCO Setaflash Tester	1991	LM	4
Petroleum Systems Services	2005	LM	4
Flow-Injection Analyzers (2):			
Bran-Leubbe	2002	LM	4
Lachat 8500	2007	LM	4
Ion Chromatographs (4)			
Dionex 2000i with Peaknet Data Systems	1988	LM	3
Dionex DX-120 with Peaknet Data System	1998	LM	3
Dionex ICS-2500 with Chromchem Data System	2002	LM	3
Dionex ICS-2000 with Chromchem Data System	2006	LM	3
Ion Selective Electrode Meters (5)			
Fisher Scientific Accument Model 50	1997	LM	6
Fisher Scientific Accument Model 25	1993	LM	6
Fisher Scientific Accument Model 20	2000	LM	6
Orion Model 920A	1990	LM	6
Corning pH/ion Meter Model 135	1992	LM	6
Microscope - Olympus	1988	LM	1
Muffle Furnace- Sybron Thermolyne Model F-A1730	1991	LM	15
pH Meters (2):			
Fisher Scientific Accument Model 20	1993	LM	6
Fisher Scientific Accument Model AR25	2005	LM	6

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY (continued)			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Shatter Box - GP 1000	1989	LM	5
Sieve Shakers (2):			
CE Tyler - Portable RX 24	1990	LM	5
WS Tyler - RX 86	1991	LM	5
Thomas-Wiley Laboratory Mill, Model 4	1989	LM	7
Total Organic Carbon (TOC) Analyzers (2)			
Coulemetrics Model 5012	1997	LM	3
O-I Corporation Model 1010	2002	LM	3
Total Organic Halogen (TOX) Analyzers (3):			
Mitsubishi TOX-Sigma	1995	LM	4
Mitsubishi TOX-100 (2)	2001	LM	4
Turbidimeter - Hach Model 2100N	1996	LM	8
UV-Visible Spectrophotometers (3):			
Hitachi 100-40 Single Beam	1986	LM	5
Beckman-Coulter DU520	2005	LM	5
Perkin Elmer Lambda 25	2008	LM	5
Vacuum Pumps (2):			
Welch Duo-Seal Model 1376	1990	LM	13
Busch R-5 Series Single Stage	1991	LM	13
Water Baths/Incubators (6):			
Hach Model 15320 Incubator	1986	LM	15
Precision Model L-6 (2)	1989, 1990	LM	15
VWR 1540	1991	LM	15
Fisher 11-680-626M Incubator	1992	LM	15
Fisher Isotemp Incubator	2001	LM	15

METALS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (6)			
Mettler AE 200 analytical balance	1990	MM	12
Various Mettler, Sartorius, and Ohaus models (5)	1988	MM	12
Atomic Absorption Spectrophotometers (5):			
Varian SpectrAA Zeeman/220 AA w/Data Systems (2)	2000	LM	3
CETAC Mercury Analyzer	2000	LM	2
Perkin Elmer AAnalyst 200 Flame AA	2005	MM	2
Atomic Fluorescence Spectrophotometer			
Brooks-Rand Model III (2)	1996, 2005	LM	3
Leeman Mercury Analyzer (1)	2006	LM	2
Centrifuge - IEC Model Clinical Centrifuge	1990	LM	12
Drying Oven - VWR Model 1370F	1990	LM	12
Freeze Dryers (2) - Labconco	1992, 2006	LM	5
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) (3)			
Thermo Jarrell Ash Model 61E	1988	LM	4
Thermo Jarrell Ash, Model IRIS	2000	MM	4
Thermo Scientific Model iCAP 6500	2007	MM	3
Inductively Coupled Plasma Mass Spectrometers (ICP-MS):			
VG Excell	2001	MM	3
Thermo X-Series	2006	MM	2
Muffle Furnace - Thermolyne Furnatrol Model 53600 (2)	1991, 2005	LM	5
Shaker - Burrell Wrist Action Model 75	1990	LM	12
TCLP Extractors (3)	1989, 2002	LM	5

SEMIVOLATILE ORGANICS SAMPLE PREPARATION LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (4)			
Mettler PM480, AE166, BB300	1999 - 2005	MM	18
OHaus EP613	2006	MM	18
Centrifuge - Sorvall Model GLC-1	1988	LM	18
Drying Ovens (2)			
Fisher Model 655G	1991	LM	18
VWR Model 1305U	1999	LM	18
Evaporators (14):			
Organomation N-Evap (7)	1989-98, 2001, 2006	LM	18
Organomation S-Evap (7)	1989-1991, 2006	LM	18
Extractor Heaters: Lab-Line Multi-Unit Models for Continuous Liquid-Liquid and Soxhlet Extractions (102)	1987-1992, 2007	LM	12
Extractors (52):			
Branson Model 450 Sonifier (2)	1991	LM	6
Tekmar Sonicator	1994	LM	6
Fisher Scientific Sonicator	1994	LM	6
Soxhtherm (48)	2000, 2008	LM	8
Extractors, TCLP (10):			
Millipore TCLP Zero Headspace Extractors (10)	1987-1992	LM	2
TCLP Extractor - Tumbler (12 position)	1989	LM	2
Gel Permeation Chromatography (GPC) (5)			
ABC single column (3)	1998, 1999, 2007	LM	4
ABC Autoprep 1000	1995	LM	4
J2 Scientific	2005	LM	4
Muffle Furnace - 4	1994-2006	LM	4
Solid Phase Extractors (8) – Horizon SPE-Dex 4790	2003, 2006	LM	4
Ultrasonic Water Bath – VWR 550D	2007	LM	18
Vacuum Pump – Edwards	1992	LM	8

GC SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler AT 250	1989	MM	7
Chromatography Data Systems (12)			7
HP Enviroquant (8)	1994-2002	LM	
Thruput Target (4)	1998-2000	LM	
Gas Chromatographs (11):			
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual ECD Detectors (4)	1990 – 1995	LM	7
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FPD Detectors	1991	LM	7
Agilent 6890 GC with Agilent 7683 Autosampler and Dual ECD Detectors (5)	2001, 2005, 2007	LM	7
Agilent 6890 GC with Agilent 7683 Autosampler and Dual FPD Detectors	2003	LM	7
Agilent 7890A Dual ECD Detectors Agilent 7683B autosampler	2008	LM	7

GC/MS SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Accelerated Solvent Extractor - Dionex ASE 200	1996	LM	5
HP Enviroquant Chromatography Data Systems (9)	1994-2002	LM	5
Gas Chromatograph: Hewlett-Packard 5890 with HP 7673 autosampler and FID Detector	1994	LM	5
Semivolatile GC/MS Systems (9):			
Agilent 6890/5973 with ATAS Optic2 LVI and HP 7673 Autosampler (2)	1997, 2001	LM	5
Agilent 5890/5970 and HP 7673 Autosampler	1990	LM	5
Agilent 5890/5970 with ATAS Optic2 LVI and HP 7673 Autosampler	1994	LM	5
Agilent 5890/5972 with ATAS Optic2 LVI and HP 7673 Autosampler (3)	1993, 1994, 1998	LM	5
Agilent 6890/5973 with ATAS Optic3 LVI and 7683 Autosampler	2004	LM	
Agilent 6890/5973 with Agilent PTV Injector and 7683 Autosampler	2007	LM	4
Semivolatile GC/MS/MS – Waters Quattro Micro GC Micromass with Agilent 6890, Agilent PTV Injector, 7683B Autosampler	2008	MM	1

PETROLEUM HYDROCARBONS GC/HPLC LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler BB240	1994	MM	6
Aspirator pump – GAST	2004	LM	6
Drying Oven - Fisher Model 630F	1991	LM	6
Evaporator - Organomation N-Evap	1990	LM	6
HP Enviroquant Chromatography Data Systems (8)	1994-2002	LM	6
Gas Chromatographs (6):			
Hewlett-Packard 5890 Series II with PID/PID/FID(2)	1991	LM	4
EST-ENCON Purge and Trap Concentrator	1991	LM	4
Dynatech Archon 5100 Autosampler	1992	LM	4
Hewlett-Packard 5890 GC with HP 7673 Autosampler and FID Detector	1995	LM	4
Agilent 6890 with Dual FID Detectors and Agilent 7873 Autosampler (3)	2001, 2005	LM	4
High-Performance Liquid Chromatographs (2):			
HP 1090M Series II with Diode Array UV Detector	1999	LM	4
HP 1050/1100 Series with Fluorescence & Diode Array UV Detectors	2004	LM	4
High-Performance Liquid Chromatograph/Mass(2) Spectrometer - Thermo Electron TSQ Quantum LC/MS/MS and Autosampler	2005	MM	2
API 5000 LC/MS/MS and SIL-20AC Autosampler	2008	MM	2

VOLATILE ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler PE 160	1989	MM	5
Fisher Vortex Mixer	1989	LM	5
HP Enviroquant Chromatography Data Systems (10)	1994-2002	LM	5
Drying Ovens (2):			
Narco 420	1989	LM	5
VWR 1305 U	1991	LM	5
Sonic Water Bath - Branson Model 2200	1989	LM	5
Volatile GC/MS Systems (7):			
Agilent 5890/5970	1989	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 5890/5971	1991	LM	5
Tekmar 3000 Purge and Trap Concentrator	2001	LM	5
Dynatech ARCHON 5100 Autosampler	1995	LM	5
Agilent 5890/5972A	1993	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 6890/5973	2001	LM	5
Tekmar 3100 Purge and Trap Concentrator	2001	LM	5
Varian Archon Autosampler	2001	LM	5
Agilent 6890/5973	2005	LM	5
Tekmar Velocity Purge and Trap Concentrator	2005	LM	5
Tekmar Aquatech Autosampler	2005	LM	5
Agilent 6890/5973 (2)	2007	LM	5
Tekmar 3000 Purge and Trap Concentrator	2007	LM	5
Varian Archon 5100 Autosampler	2007	LM	5

DRINKING WATER ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler BB300	1991	MM	2
Extractors (10) – Horizon SPE-DEX Solid Phase Extractor	2003/2008	LM	2
Aglinet Enviroquant Chromatography Data Systems (2)	2003	LM	2
Varian Saturn Chromatography Data System	2003	LM	2
Evaporator - Organomation N-Evap	2003	LM	2
Agilent 1100 HPLC w/post-column derivitization: UV/Fluoescence detectors Pickering PCX-5200 Post-column derivitization unit	2003 2003 2003	LM LM LM	2 2 2
Agilent 6890N GC/Dual ECD system w/ autosamplers Agilent 7890 GC/Dual ECD w/autosamplers	2003 2008	LM LM	2 2
Varian Ion trap GC/MS: Varian 3800 GC w/CP8400 autosampler Varian Saturn 2100T mass spectrometer	2003 2006 2003	LM LM LM	2 2 2
Thremo Ion Trap GC/MS w/TriPlus autosampler	2008	LM	2

Metals Method Development Laboratory			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Perkin-Elmer ICP/MS Elan 9000 w/ Perkin-Elmer AS-93+ Autosampler	2008	LM	2
Perkin-Elmer Series 200 IC	2008	LM	2
Brooks Rand III Atomic Fluoresence Spectrophotometer - 2	2008	LM	2
Oriel Atomic Fluoresence Spectrophotometer – Lab Designed	2008	LM	2
Balances - 4	2008	LM	2
Ovens - 2	2008	LM	2
Buck AA Spectrophotometer Model 205	2008	LM	2
Forma Scientific Bio Freezer	2008	LM	2
Digital Shaker SK-71	2008	LM	2

AUTOMATED DATA PROCESSING EQUIPMENT			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
1-WAN: LIMS Sample Manager using Oracle 10g DBMS running on Redhat Advanced Server 3.0 (Linux) platform connected/linked on a frame relay WAN environment	1994-2004	LM	NA
1 - Network Server Pentium 4 class, 1 for Reporting and Data Acquisition running Windows 2003 Advanced Server, 1 for Applications running Windows 2003 Advanced Server. Data acquisition capacity at 65GB with redundant tape and disk arrays.	2004	LM	NA
Approximately 50+ HP and Dell Laserjet printers (various types including models III, 4, 5, 8150, 4000, 4050, 4250, 8150, 1720dn, W5300)	1991 - 2007	LM	NA
Approximately 180 Gateway/Dell PC/Workstations running Windows 2000/XP on LAN connected via 10BT/100BT and TCP/IP for LIMs Terminal Emulation	1993 - 2004	LM	NA
Microsoft Office 2003 Professional as the base application for all PC/Workstations. Some systems using Office 2000/97.	1996 - 2004	LM	NA
E-Mail with link to SMTP for internal/external messaging. Web mail via Outlook Web Access interface. Microsoft Outlook 2003.	1994 - 2006	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Facsimile Machines - Brother 4750e (2); Brother SuperG3 (1); Canon CFX-L4000 (1)	1991 - 2007	LM	NA
Copiers/Scanners: Konica BizHub 420 (1), BizHub 600 (1), BizHub 920 (2), BizHub Pro 1050 (3). The 920s and 1050s are accessible via LAN for network scanning.	2000 - 2007	LM	NA
Dot Matrix Epson FX-880, LQ-1050, LX-300	1991 - 2004	LM	NA
Thruput, MARRS, Stealth, Harold, Blackbird, EDDGE, StarLIMS reporting software systems.	1998 - 2004	LM	NA

NA: Not applicable. This equipment administered by IT staff but may be used by all staff.

APPENDIX D

PREVENTIVE MAINTENANCE PROCEDURES

Instrument	Activity	Frequency
Refrigerators and Coolers	Record temperatures	Daily
	Clean coils	Annually
	Check coolant	Annually or if temperature outside limits
Vacuum Pumps	Clean and change pump oil	Every month or as needed
Fume Hoods	Face velocity measured	Quarterly
	Sash operation	As needed
	Change filters	Annually
	Inspect fan belts	Annually
Ovens	Clean	As needed or if temperature outside lim.
	Record temperatures	Daily, when in use
Incubators	Record temperatures	Daily, morning and evening
Water Baths	Record temperatures	Daily, morning and evening
	Wash with disinfectant solution	When water is murky, dirty, or growth appears
Autoclave	Check sterility	Every month
	Check temperature	Every month
	Clean	When mold or growth appears
Analytical Balances	Check alignment	Before every use
	Check calibration	Daily
	Clean pans and compartment	After every use
Dissolved Oxygen Meter	Change membrane	When fluctuations occur
pH probes	Condition probe	When fluctuations occur
Fluoride ISE	Store in storage solution	Between uses
Ammonia ISE	Store in storage solution	Between uses
UV-visible Spectrophotometer	Wavelength check	Annually
Total Organic Carbon Analyzers	Check IR zero	Weekly
	Check digestion/condensation vessels	Each use
	Clean digestion chamber	Every 2000 hours, or as needed
	Clean permeation tube	Every 2000 hours, or as needed
	Clean six-port valves	Every 200 - 2000 hours, or as needed
	Clean sample pump	Every 200 - 2000 hours, or as needed
	Clean carbon scrubber	Every 200 - 2000 hours, or as needed
	Clean IR cell	Every 2000 - 4000 hours, or as needed

Instrument	Activity	Frequency
Total Organic Halogen Analyzers	Change cell electrolyte	Daily
	Change electrode fluids	Daily
	Change pyrolysis tube	As needed
	Change inlet and outlet tubes	As needed
	Change electrodes	As needed
Flow Injection Analyzer	Check valve flares	Each use
	Check valve ports	Each use
	Check pump tubing	Each use
	Check light counts	Each use
	Check flow cell flares	Quarterly
	Change bulb	As needed
	Check manifold tubing	Each use
	Check T's and connectors	Each use
Ion Chromatographs	Change column	Every six months or as needed
	Change valve port face & hex nut	Every six months or as needed
	Clean valve slider	Every six months or as needed
	Change tubing	Annually or as needed
	Eluent pump	Annually
Atomic Absorption Spectro-photometers - FAA and CVAA	Check gases	Daily
	Clean burner head	Daily
	Check aspiration tubing	Daily
	Clean optics	Every three months
	Empty waste container	Weekly
Atomic Absorption Spectro-photometers - GFAA	Check gases	Daily
	Check argon dewar	Daily
	Change graphite tube	Daily, as needed
	Clean furnace windows	Monthly
ICP - AES	Check argon dewar	Daily
	Replace peristaltic pump tubing	Daily
	Empty waste container	Weekly
	Clean nebulizer, spray chamber, and torch	Every two weeks
	Replace water filter	Quarterly
	Replace vacuum air filters	Monthly

Instrument	Activity	Frequency

Instrument	Activity	Frequency
ICP - MS	Check argon dewar	Daily
	Check water level in chiller	Daily
	Complete instrument log	Daily
	Replace peristaltic pump tubing	Daily
	Clean sample and skimmer cones	As needed
	Clean RF contact strip	As needed
	Inspect nebulizer, spray chamber, and torch	Clean as needed
	Clean lens stack/extraction lens	As needed
	Check rotary pump oil	Monthly
	Change rotary pump oil	Every six months
Gel-Permeation Chromatographs	Clean and repack column	As needed
	Backflush valves	As needed
High Pressure Liquid Chromatographs	Backflush guard column	As needed
	Backflush column	As needed
	Change guard column	As needed when back pressure too high
	Change column	Annually or as needed
	Change in-line filters	As needed
	Leak check	After column maintenance
	Change pump seals	As needed
	Change pump diaphragm	Annually
	Clean flow cell	As needed
	Fluorescence detector check	Daily
	Diode array absorbance check	Daily
Gas Chromatographs, Semivolatiles	Check gas supplies	Daily, replace if pressure reaches 50psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Change injection port liner	Weekly or as needed
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Clean FID	Weekly or as needed
	Clean ECD	Quarterly or as needed
	Leak test ECD	Annually

Instrument	Activity	Frequency
Gas Chromatograph/Mass Spectrometers, Semivolatiles	Check gas supplies	Daily, replace if pressure reaches 50psi
	Change in-line filters	Annually or as needed
	Change septum	Daily, when in use
	Change injection port liner	Weekly or as needed
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean source	As needed when tuning problems
	Change pump oil	As specified by service specifications
Purge and Trap Concentrators	Change trap	Every four months or as needed
	Change transfer lines	Every six months or as needed
	Clean purge vessel	Daily
Gas Chromatographs, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Clean PID lamp	As needed
	Clean FID	As needed
	Change ion exchange resin	Every 60 days
	Replace nickel tubing	Quarterly or as needed
Gas Chromatograph/Mass Spectrometers, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Annually or as needed
	Change septum	Daily
	Clip first foot of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean jet separator	As needed
	Clean source	As needed when tuning problems
	Change pump oil	As specified by service specifications

APPENDIX E

CORPORATE POLICY STATEMENTS

Columbia Analytical Services™

Policy for Data Review and Validation

May 2009
Effective July 1, 2009

The purpose of this policy is to identify the requirements for performing data review and validation prior to releasing data and reports to customers of Columbia Analytical Services. It is a requirement of NELAC (TNI) quality system standards and Department of Defense (DoD) agencies to have data review procedures established.

This policy is applicable to the review of raw and reported data generated in all laboratories. Specific data review and validation processes or logistics may vary somewhat from facility to facility, or vary for data generated using different methodologies however; the policies described here are to be followed. The documentation practices should be consistent within the facility. Automated validation processes are encouraged, but must be sufficiently described in an SOP.

In general, the data review and validation practices used at each facility will meet the requirements of NELAP quality system standards, the DoD Quality System Manual (QSM), and ISO 17025. Specific data review and validation policies are as follows:

1. Each laboratory facility will have a written and approved standard operating procedure (SOP) for conducting data review/validation that meets the standard CAS requirements for administrative SOPs. The SOP will list details of data review practices for the facility. The SOP will also give a detailed explanation of the review documentation procedures for each type of data.

2. Data review will be performed by qualified personnel who have documented training on either the analysis itself or training specific to the data review SOP. Personnel preparing reports who may do some level of clerical review or proofreading do not need technical knowledge of the test, but must be knowledgeable of reporting systems and requirements.

3. All data will be reviewed by a minimum of two persons. Data generated or reported by one person may not be released without another person's review.

4. However defined, one review (typically a "primary" technical review) must focus on the validity of the analysis and raw data generated, the technical accuracy and correctness of the analysis (the analytical procedure is in control), use of valid and approved procedures and methods, and interpretation of sample results.

5. The secondary review will be performed by someone other than the technical reviewer. The secondary review will make the same assessments as the primary reviewer, and check the interpretations, data manipulations, and decisions made by the primary reviewer. Additionally, the secondary reviewer will review the outputs from the initial review to the raw data. This includes such things as data processing results/outputs, calculations, runlogs, bench sheets, QC analyses, etc. The secondary review verifies the completeness and validity of the data to be reported.

6. All client-ready final reports will be reviewed in the format, and as presented to, the client; either by analysis fraction or in their entirety. This review will include verification of the accurate and correct reporting of sample and QC results; including accurate translation of results from data to report forms, report format, use of qualifiers and flags, and method citations. This review will also include verification of the correct project information; such as client name, project name, sample I.D.s, etc. The report review should ensure that the report is error-free and contains no inconsistencies. For upper tier deliverables, this review will verify that all deliverables are included in the report package.

7. The Project Manager will review all complete reports prior to signing the report and submitting to the client. The review of the reported data will focus on the following items:
    a. Consistency with client, contract, and/or project specifications.
    b. Acceptability of any data qualifiers or footnotes.
    c. Accuracy and completeness of explanations or discussion in the report cover letter or case narrative.
    d. As needed depending on the scope of testing, an additional level of technical review of all data generated.
    e. A general overview of the completed service request file with respect to overall reasonableness, and if available, with historical project information.

8. Data review must be documented. Persons performing data and report review must sign (or initial) and date the applicable data reviewed. Checklists or review summaries should be used for guidance and documentation. Documentation processes must be described in the laboratory SOP.

Lee Wolf, Corporate Director of Quality Assurance — Date: 5-5-09

Steve Vincent, President — Date: 5-5-09

Columbia Analytical Services

Policy for Conducting Research, Method Development, and Method Investigations

December 2009

Columbia Analytical Services (CAS) often develops test procedures internally by conducting research and development or method development based on published procedures. This type of testing may not fall under common laboratory regulations which describe benchmarks, or minimum requirements, for procedure development and implementation. Also, it may be necessary at certain times to conduct investigations into the quality of existing methods. Therefore, a policy is necessary to identify and establish those minimum requirements.

The purpose of this Policy is to identify the CAS requirements for performing internal research and subsequent method development, performing method development from published references, and performing investigations into method performance.

For the purpose of this policy, the following Definitions are provided:

Research and development (R&D) – The practice of independently evaluating analytical options and procedures and applying them to a sample analysis challenge; resulting in an internally developed analysis method. For this policy, R&D is limited to that performed by CAS personnel.

Method development – The practice of implementing a CAS analysis procedure based on published references.

Method investigation – For the purpose of this policy, this is defined as the evaluation of major changes in methodology outside the scope of published methods or SOPs. This is generally done to improve method performance or troubleshoot a significant analytical problem; and done outside of the routine maintenance, troubleshooting, and nonconformance/corrective action process.

The intent of this policy is to ensure that CAS R&D, method development, and method investigations are performed in an unbiased manner, ensure data integrity, use common scientific practices; and ensure that these activities are peer reviewed.

General Provisions

- When conducting any of the activities covered by this policy, employees will follow standard CAS procedures for maintaining documentation and analysis records.
- Initial and final review of statements, plans, and summaries will be done by two persons; the applicable Technical Director (TD) and the Laboratory Director (LD). If the TD is the LD, then a Peer will conduct the second review.
- Once development is concluded, the adoption of Standard Operating Procedures (SOPs), conducting personnel training, etc., will be done following routine CAS QA protocols.

Research and Development

When conducting research on new analyses and developing in-house procedures not based on reference methods or published methods, the research and development effort will include the following components:

1) There will be a written *Development Statement* detailing the intent of the research and development effort. This will state the purpose of the work, the resources and references expected to be used, the experimentation that will be performed, and the anticipated result. The following items will be included in the statement:
   a) Equipment to be used.
   b) Quality Control measures to be incorporated into the analysis.
   c) Method Performance (validation) measures to be taken and expectations.
2) There will be an initial internal review and acceptance of the statement by the Technical Director and the Laboratory Director.
3) The person leading a R&D effort will gather information, references, and resources as described in the Development Statement and document those resources.
4) The experimentation will be performed and documented.
5) Once data is collected, it will be interpreted objectively using common assessments of bias and precision. Tests for false negative and false positive results will be used as well as measurements of accuracy and precision.
6) The developer will draw conclusions, and if successful, summarize the results in a brief R&D summary.
7) The summary report will include a documented approval by the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
8) Following approval, an SOP will be written for subsequent implementation.

Method Development

When developing and implementing new methods based on reference or published methods, the method development effort will include the following components:

Non-certified (nor certifiable) methods

1) There will be a written *Development Statement* detailing the method development effort. This will state the purpose of the work, the reference method, references expected to be used, the experimentation that will be performed, and the anticipated result. The following items will be included in the statement:
   a) The reference method being implemented and the application(s).
   b) Equipment to be used.
   c) Quality Control measures to be incorporated into the analysis.
   d) Method Performance (validation) measures to be taken and expectations.
   e) Modifications to the reference method.
2) There will be an initial internal review and acceptance of the statement by the applicable Technical Director and the Laboratory Director.

3) The experimentation will be performed and documented.
4) Once data is collected, it will be interpreted objectively using common assessments of bias and precision. Tests for false negative and false positive results will be used as well as measurements of accuracy and precision.
5) The developer will draw conclusions, and if successful, summarize the results in a brief method development summary.
6) The summary report will include a documented approval by the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
7) Following approval, an SOP will be written for subsequent implementation on the stated applications.

Certified (certifiable) methods

1) There will be a written *Experimental Plan* detailing the method development effort. This will state the method being implemented, references expected to be used, the experimentation that will be performed, and the anticipated result. The following items will be included in the Plan:
   a) The reference method being implemented.
   b) Equipment to be used.
   c) Quality Control measures to be incorporated into the analysis.
   d) Method Performance (validation) measures to be taken and expectations. This will include method and certification requirements for accuracy and precision, sensitivity, selectivity, calibration/linear range, etc. For methods where NELAC accreditation is being pursued, the requirements of the NELAC Standard (2003 Standard, Quality Systems section 5, Appendix C.3) will be met.
   e) Modifications to the reference method.
2) There will be an initial internal review and acceptance of the Plan by the applicable Technical Director and the Laboratory Director.
3) The method will be set up and run following the procedural steps of the method and the Plan; and will be documented.
4) Once data is collected, it will be interpreted objectively using common assessments of bias and precision. Tests for false negative and false positive results will be used as well as measurements of accuracy and precision.
5) The developer will draw conclusions, and if the results meet the method performance criteria in the method and/or Experimental Plan, the results will be summarized in a brief method development summary.
6) The summary report will include a documented approval the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
7) Following approval, an SOP will be written for subsequent implementation.

Method Investigations

1) There will be a written *Investigation Statement* detailing the method investigation effort. This will state the purpose of the investigation, the CAS procedure, the targeted problem, the experimentation that will be performed, and the desired improvement result. The following items will be included in the statement:
   a) The CAS procedure being investigated and the equipment used.
   b) A brief discussion of the problem, the solutions being investigated, and the impact on method compliance and data quality.
   c) The experimentation used to perform the investigation.
   d) The Method Performance (validation) measures that will be taken to re-establish conformity to QA/QC criteria.
2) There will be an initial internal review and acceptance of the statement by the applicable Technical Director and the Laboratory Director.
3) Once data is collected, it will be interpreted objectively using the assessments applicable to that analysis and CAS SOP.
4) The investigator will draw conclusions, and if the results meet the method performance criteria in the method and SOP, the results will be summarized in a brief method investigation summary.
5) The summary report will include a documented approval by the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
6) Following approval, the CAS SOP will be revised to implement the changes to procedure.

Documentation

The developer or investigator will generate the written Development or Investigation statements, or Experimental Plan, and provide them for initial review prior to beginning experimentation and data collection. The initial review and acceptance of the Statement will be documented. The laboratory QA PM will keep this documentation on file.

The developer or investigator will generate the written summary report and validation package, and will submit supporting data for review. The approval of the development or investigation (and SOP changes) will be documented and the laboratory QA PM will keep this documentation on file.

Steve Vincent

Steve Vincent, President/CEO

12-15-09

Date

Lee Wolf

Lee Wolf, Chief Quality/Ethics Officer

12-15-09

Date

Columbia Analytical Services™

Policy for Standards and Reagents Expiration Dates

September 2009
Effective September 28, 2009

The purpose of this policy is to state the standardized requirements for assigning expiration dates to standards and reagents used in the laboratories of Columbia Analytical Services. It is a requirement of NELAP Quality System standards, the DoD Quality System Manual (QSM), and ISO 17025 to have written protocols to ensure the use of standards and reagents of appropriate quality. Additionally, documentation of the expiration date of reagents and standards is required. This policy is intended to meet the requirements of NELAC, DOD, and ISO 17025.

This policy is applicable to all purchased and prepared standards and reagents used by the laboratory to generate reported data. This includes raw (neat) materials, stock, intermediate, working, and calibration standards and/or reagents. This does not include solvents and acids.

In general, the expiration date is the date after which a standard or reagent shall not be used. It is either the date assigned by the manufacturer, the date (duration) specified by the applicable reference method, or it is a date assigned by the laboratory under this policy.

General Policies:

1. All standard and reagent expiration dates/periods shall be listed in the applicable laboratory SOP.

2. When establishing an expiration date, the following hierarchy will be used:

   - If the cited analytical method specifies the expiration date/period, that date shall be used.
   - If the cited analytical method does not specify the expiration date/period, then the date assigned by the manufacturer will be used.
   - If the cited analytical method does not specify the expiration date/period, and an expiration date is not assigned by the manufacturer, then the laboratory will assign the expiration date according to the CAS Standardized Expiration Dates tables below.

CAS Expiration Dates for Reagents	
Chemical	Expiration Date
Purchased neat reagents	5 years after receipt
Inorganic reagent solutions	1 year from preparation or receipt
Organic reagent solutions	6 months from preparation or receipt

CAS Expiration Dates for Standards		
Chemical	Expiration Date	
Purchased neat standards	5 years after receipt	
Inorganic stock standard solutions	1 year from preparation or receipt	
Inorganic secondary, intermediate, or working standard solutions	6 months from preparation or receipt	
Purchased semivolatile organic stock standard solutions	1 year from receipt	
Prepared semivolatile organics stock standards	1 year from preparation	
Semivolatile organic secondary, intermediate, or working standard solutions	6 months from preparation or receipt	
Purchased volatile organics stock standards – **unopened** ampules	1 year from receipt	
Purchased volatile organics stock standards – **opened** ampules	≤2000 mg/L >2000 mg/L	1 month after opening 3 months after opening
Prepared volatile organics stock standards	1 year from preparation	
All volatile organics secondary, intermediate, or working standards* * note: common 'gases' standards and standards used for calibration should not be older than 7 days	≤20 mg/L >20 and ≤200 mg/L >200 mg/L	7 day expiration date 1 month expiration date 3 month expiration date
Dioxin/Furan and PCB stock standards	5 years from receipt	
Dioxin/Furan and PCB working standards	1 year from preparation or receipt	
Derivatized (prepared) semivolatile organics standard solutions	1 year from date of derivatization	

3. The expiration date of a prepared reagent or standard cannot exceed the expiration date of the starting material, with the exception of standards prepared via in-lab derivatization to yield a different compound. The expiration date of a reagent or standard cannot be extended by preparing a dilution of it. For example, a purchased standard has an expiration date of July 15, 2009. A standard prepared on February 20, 2009 from this purchased standard would ordinarily have an expiration date of six months (namely, 8/20/2009), but since the purchased standard expires before six months, the prepared standard would be assigned an expiration date of July 15, 2009.

4. A multicomponent prepared reagent or standard will be assigned an expiration date not to exceed the expiration date of any of the components' expiration date. For example, a prepared standard is made from purchased standard A (with an expiration date of August 5, 2009) and from purchased standard B (with an expiration date of December 15, 2009). Consequently, the prepared standard will have an expiration date of August 5, 2009.

5. The stability and concentration of the reagent or standard are to be taken into account when assigning the expiration date. Certain solutions, depending on use and storage, may have shorter usable life time than defined by the method, manufacturer, or this policy; and should be assigned expiration dates accordingly. Reagents and standards must be stored under conditions specified by the test method and outlined in the analytical SOP.

6. Expiration dates can be extended under the following conditions:
   - A new, replacement reagent or standard is not readily available from vendors and,
   - The cited analytical method does not specify the expiration date/period and,
   - The material has been stored under conditions specified by the analysis method and outlined in the analytical SOP and,
   - The material is not reactive, volatile, or prone to degradation under the specified storage conditions and,
   - The suitability of the material is verified by the laboratory as follows, under the same valid analysis conditions used for sample analysis, and meet the following criteria:

     a. For reagents:
        i. Perform a blank and LCS pair of analysis three times using three different subaliquots of the reagent.
        ii. Each LCS result must be within the specified control limits for the test.
        iii. The %RSD for the three LCS's must be <10%.
        iv. Each blank result must be < 1/2MRL for every compound to be reported from subsequent analysis.
     b. For standards:
        i. Analyze three separate dilutions of the standard at a concentration near the midpoint of the calibration range. (Note that standards below this concentration cannot be re-verified).
        ii. The average result must be within ± 5% of the original true value.
        iii. The %RSD for the three results must be <10%.

     If these conditions and criteria are met and documented, the material may be assigned a new expiration period the same as newly prepared material.

Lee Wolf, Corporate Director of Quality Assurance — Date: 9-10-09

Steve Vincent, President — Date: 9-15-09

Policy for the Use of Accreditation Organization Names, Symbols, and Logos

September 2009

Effective October 1, 2009

The purpose of this policy is to state Columbia Analytical Services' (CAS) requirements and restrictions for the company use of the name, symbols, and logos of accreditation organizations. In general, the names, symbols, and logos used by these organizations are the property of the organization. Therefore, it is a policy that CAS will comply with the requirements and policies of the organizations that accredit our laboratories.

The NELAC Institute (TNI): The TNI Board of Directors approves and oversees the use of TNI logos and marks (TNI, NELAC, NELAP) by programs, members, and other entities. In consideration that CAS is a member of TNI, CAS will abide by the following TNI policy and be subject to the TNI Consequences of Misuse.

All persons and entities that use or reproduce TNI logos and marks:

1. *Shall restrict access to them by unauthorized parties.*
2. *Shall use them only for purposes and activities authorized by the TNI Board of Directors.*[a]
3. *Shall endeavor to avoid statements in relation to their use that the TNI Board of Directors may consider misleading or unauthorized.*
4. *May not imply endorsement or approval by TNI in communication media such as the Internet, documents, brochures, or advertising without the expressed consent of the TNI Board of Directors.*
5. *May not imply an association or partnership with TNI when such an arrangement has not been authorized by the TNI Board of Directors.*

[a] Authorized uses and activities are listed in the 2003 NELAC Standard, Section 6.8

American Association for Laboratory Accreditation (A2LA): CAS will comply with A2LA policy *P101 – Reference to A2LA Accredited Status – A2LA Advertising Policy*[b].

- CAS will only use the A2LA logo and symbol/phrase "A2LA Accredited" at individual CAS laboratory locations which have demonstrated to be in compliance with A2LA quality system requirements for the applicable A2LA accreditation program (e.g. Testing Laboratory).
- The "A2LA Accredited" symbol will not be used by a CAS laboratory that is not A2LA accredited and the symbol will not be used by a CAS laboratory that has only applied for accreditation.

- When promoting A2LA accreditation, CAS will follow the requirements of the A2LA policy.
- Where the "A2LA Accredited" symbol is used to endorse results on reports, it will always be accompanied by the A2LA certificate number and an indication of the type of laboratory (i.e., testing laboratory).

[b] The A2LA policy can be found at http://www.a2la.org/policies/A2LA_P101.pdf

International Organization for Standardization (ISO): ISO does not perform assessments and therefore is not a certification or accreditation organization. ISO is a standards development organization and compliance with an ISO standard does not imply ISO endorsement. ISO's statement on the use of the name and logo is listed below, and can be found at the following URL: http://www.iso.org/iso/support/name_and_logo.htm ISO has also provided a guide for how to publicize certification to an ISO standard: http://www.iso.org/iso/publicizing2005-en.pdf

Use of ISO's name®

Within the context of international standardization or related activities (such as consultancy, training or conformity assessment including certification) "ISO" (or "iso") is the short name of the International Organization for Standardization. The name is registered within this context as the sole property of ISO and the Organization will protect its name on behalf of all ISO's members - the national standards institutes of some 150 countries. In particular, ISO will not authorize the use of the name "ISO" (or "iso") by any organization other than its members in Internet domain names, names of Web sites, trademarks, companies / organizations, products, etc. Such use could mislead third parties into believing that the domain name / Web site / trademark / company / organization / product concerned represents ISO, or has been approved or authorized to act on behalf of ISO or belongs to ISO.

Therefore, ISO will take whatever actions it considers necessary to prevent the misuse of its name.

Use of ISO's logo®

The ISO logo is a registered trademark. Unless authorized by ISO, use of its logo is prohibited. Notably, ISO will not allow its logo to be used in connection with conformity assessment activities. These include the certification of management systems, products, services, materials or personnel, even when these certifications attest conformity to an ISO standard, such as one of the ISO 9000 or ISO 14000 series. Examples of unacceptable use of the ISO logo would include use on products, in publications, on Internet sites, in marketing materials, advertisements and company letterheads.

Allowing the ISO logo to be used would give the false impression that ISO carries out certification activities, or has approved or authorized the organization using its logo. These activities are not business functions of ISO.

ISO is not an auditor, assessor, registrar, or certifier of management systems, products, services, materials or personnel, nor does ISO endorse any such activities performed by other parties. ISO develops International Standards but does not operate any schemes for assessing conformance with them.

Therefore, ISO will take whatever actions it considers necessary to prevent the misuse of its logo.

The organizations specifically discussed in this policy do not comprise a complete list of organizations to which the policy applies. It is reiterated that, with regards to the use of names, symbols, and logos; it is a policy that CAS will comply with the policies of the organizations that accredit our laboratories.

Lee Wolf, Corporate Director of Quality Assurance — Date: 9-21-09

Steve Vincent, President — Date: 9-21-09

Columbia Analytical Services™

Policy for Internal Quality Assurance Audits

May 2009
Effective July 1, 2009

The purpose of this policy is to identify the requirements for performing internal systems audits and data audits in the laboratories of Columbia Analytical Services. Internal audits are necessary to ensure that laboratory operations work within the quality systems and that these systems yield data of high quality. Internal audits are also necessary in order to meet certification and accreditation requirements. The internal auditing practices used at each facility will meet the requirements of NELAC quality system standards, the Department of Defense (DoD) Quality System Manual (QSM), and ISO 17025.

For systems audits, the concept of this policy is that corporate quality assurance audits will evaluate the laboratory QA systems and operation horizontally, or as an overall 'umbrella' assessment, whereas local QA audits will be 'drill down' audits focused on technical correctness and data validity. It is practical to verify related systems implementation as these audits are conducted.

For electronic data auditing, the concept is to assess critical data from high liability steps of procedures, from all applicable instruments, in a frequent manner (quarterly) so as to identify any potential problems relatively quickly. This is in contrast to performing 100% data assessment from a subset of instruments quarterly and taking a long period of time to assess all instruments.

Definitions

- System audits are audits used to evaluate quality system implementation, policies, procedures, laboratory practices, and testing activities of the laboratory.

- Data audits are used to assess reported laboratory data. This includes all data used to generate the reported results and the final report itself. These are performed as 'desk audits' of reported data packages, and supporting data if not included in the reported data.

- Electronic data audits are used to assess laboratory data that is processed, interpreted, used by the analyst in electronic format. This is generally limited to electronic chromatographic data.

    - "Critical" and "high liability" data – Data related to the tuning, calibration, calibration verification, and QC analyses for an analysis; as well as data vulnerable to improper manipulation (improper processing/reprocessing of files, clock changes, poor interpretation of control data, peak integrations, etc., as described in CAS Ethics policies).

Specific internal auditing policies are as follows:

1. A comprehensive internal audit will be conducted annually (approximately every 12 months) at each laboratory. The audit will address all elements of the quality system and will include environmental testing activities, and used to meet the annual internal audit requirements of NELAC, DoD, and ISO 17025. In general, the comprehensive audit will be conducted and lead by the Quality Assurance Director (QAD), with assistance from the laboratory Quality Assurance Program Manager (QA PM).

   The laboratory QA PM will not be required to conduct an additional comprehensive audit. While performing the system and data audits described below, the QA PM will verify ongoing implementation of many QA systems.

2. Each laboratory QA PM will conduct three technical systems audits per calendar quarter. These audits will be technically-focused audits of three different test procedures and technologies.
   a. The three procedures will be varied throughout the year such that analytical disciplines (e.g. digestion, extraction, ICP, ICP/MS, titrimetric, colorimetric, GC, GC/MS, HPLC, microbiology, etc) from all sections of the laboratory are assessed in a year (for laboratories with fewer than 12 tests performed, the same tests will be audited more than once).
   b. The audits will assess SOP and method compliance.
   c. The audits will assess the use of sound analytical techniques and practices.
   d. The audits will assess the analyst(s) training and documentation of /proficiency.
   e. The audit will assess all aspects of the test being evaluated, including sample handling/preparation, calibration, sample batching/run sequences, standards, quality control, instrument operation/maintenance, data interpretation, data review/reporting, and applicable quality assurance.

3. Each laboratory will conduct two complete hardcopy data audits per quarter. These audits will focus on data validity, accuracy, and completeness. Data audits will be performed on hardcopy raw and reported data (or electronic version of) and on a 'Service Request basis'.
   a. The audits will be performed on data generated no earlier than three months prior to the audit.
   b. Service requests are to be chosen at random to encompass various analytical disciplines of the laboratory over the course of a year.
   c. The audit will assess the validity of the laboratory procedures used to generate the results reported, from sample receipt to analysis to data reporting, and the accuracy and completeness of the final report.
   d. The audit may be used as a convenient way to assess training documentation for the analysts who performed the analyses.

4. DoD report reviews will be conducted quarterly at the frequency required by the DoD QSM.

5. Electronic data auditing

    a. Each laboratory will conduct random screening of chromatographic data using Mint Miner software (where analytical software is compatible) every quarter on every instrument on data generated that quarter.
    b. Mint Miner software will be adequately configured in order to make screening effective.
    c. Using the screening results, data files will be selected for auditing from each instrument each quarter. Two sequences will be audited, one an initial calibration and one a typical sample analysis sequence. Test methods are to be chosen at random to encompass various methods performed.
    d. The audits will focus on calibration and QC data, including the evaluation of proper processing of files, interpretation of data, peak integrations, and comparison of raw electronic data to 'interpreted' and approved data.
    e. If screening results indicate significant potential problems, additional files should be inspected. The QA PM will conduct these added audits as needed.
    f. If Mint Miner software is not compatible with instrument software, auditing will be performed manually by the QA PM by auditing the data from two sequences per quarter, including one initial calibration sequence, per instrument.

6. As with any audit, additional auditing and investigation may be necessary based on the audits performed and magnitude of findings.

7. Each laboratory facility will have a written and approved standard operating procedure (SOP) for conducting their internal audits. The SOP will include detailed procedures for technical system audits, data audits, and electronic data audits as defined in this policy. In addition to meeting the standard CAS requirements for administrative SOPs, the SOP will include details of the audit processes, use of checklists, documentation, audit reporting, corrective action, and resolution of audit findings.

Lee Wolf, Corporate Director of Quality Assurance — Date: 5-5-09

Steve Vincent, President — Date: 5-5-09

Columbia Analytical Services

CAS Quality and Ethics Policy Statement

March 2009

Columbia Analytical Services (CAS) vision is simple. We strive to be the best in everything we do. This includes ethics and professional practice where CAS is committed to the highest standards of ethical behavior and quality of its analytical testing.

Unethical behavior carries a heavy price - one that we do not want to bear. This includes loss of reputation, loss of business, civil and criminal penalties, and government and customer sanctions.

CAS is committed to excellence and superior performance in everything we do. We will not sacrifice our ethical principles in order to achieve business success. This means we will always strive to conduct business honestly and with integrity. We will always follow and obey the law of the land in which we are operating our business. We will always follow, to the best of our ability, standard operating procedures, rules and regulations that apply to our industry and specifically to our laboratory operations. Our customers, employees, suppliers and communities that we serve expect and deserve nothing less than the highest standards of conduct and compliance.

The following are the critical elements of the Quality and Ethics program at CAS.

- The Executive Management and Board of Directors of CAS sponsor and support the Quality and Ethics program through their personal commitment and by providing the necessary resources to promote this program throughout the organization.
- Chief Quality and Ethics Officer. The position is responsible for the quality and ethics program, ensures that appropriate resources are provided, reviews and recommends changes in the program, and resolves ethical and quality issues brought to management attention. This Officer reports directly to the Board of Directors Audit Committee on quality and ethics.
- Core Values. The CAS Statement of Core Values was developed internally with input from the entire company. We are committed to ensuring the integrity and quality of data, and meeting the needs of our clients, while conducting business with high ethical standards. We hold strong to the core values of Honor, Truth, and Fairness. We are committed to these values and rely on them when confronted by difficult choices.
- Ethical Code of Conduct. As a member of the American Council of Independent Laboratories (ACIL) and part of the laboratory industry, CAS subscribes to and supports the core values and ethical codes established by this industry organization.

- CAS Code of Conduct. CAS requires its employees to be introduced to and to sign the "CAS Commitment to Excellence in Data Quality" statement and to comply with standards outlined in Section 6, Employee Conduct, of our Employee Handbook. All personnel concerned with analytical testing activities within the laboratory are required to acquaint themselves with the quality documentation and to implement these policies and procedures in their work.
- Open Door Policy. Employees have the right and obligation for open communications to ask questions, seek guidance, and report incorrect practices and wrong doing without fear of retribution. As described in the CAS Open Door Policy; CAS believes in using the chain-of-command channels for this dialogue. However, if there is fear or a concern that using this approach is not appropriate, employees are free to take their concerns to the President, the Director of Human Resources, the Chief Administrative Officer, the Chief Quality Officer, or the company Ombudsman. Employees may do so without fear of retribution.
- Ombudsman Program. CAS has implemented an external ombudsman/hotline program through EthicsPoint, a phone and internet-based reporting system, to enhance communication and empower employees to promote safety, security, and ethical behavior. Employees can file a report anonymously to address issues in the workplace and to cultivate a positive work environment.
- Internal Audits. Policies are established to ensure that internal systems and data audits are conducted periodically in addition to external agency and client audits. The data audits include a detailed in-depth review of hardcopy data and electronic data to ensure compliance with the CAS Quality program and on-going data integrity.
- NELAP Accreditation. CAS management is committed to compliance with the NELAP standards. CAS maintains NELAP accreditation and as such includes quality systems documented in QA Manuals, documented procedures in Standard Operating Procedures (SOPS) and policies, and documented training for demonstration of capabilities.
- Ethics Training. CAS has the obligation to provide training to its employees with respect to company policies concerning business conduct. This includes introductory training on this, and related policies, at the time of hire; in-depth "core" training within one year of hire, and on-going refresher training on a semi-annual basis.

The CAS Quality and Ethics Program has been in place for several years. However, this is a "living" program that will change and improve as the company grows and changes.

Steve Vincent, President/CEO — Date: 3-19-09

Lee Wolf, Chief Quality/Ethics Officer — Date: 3-19-09